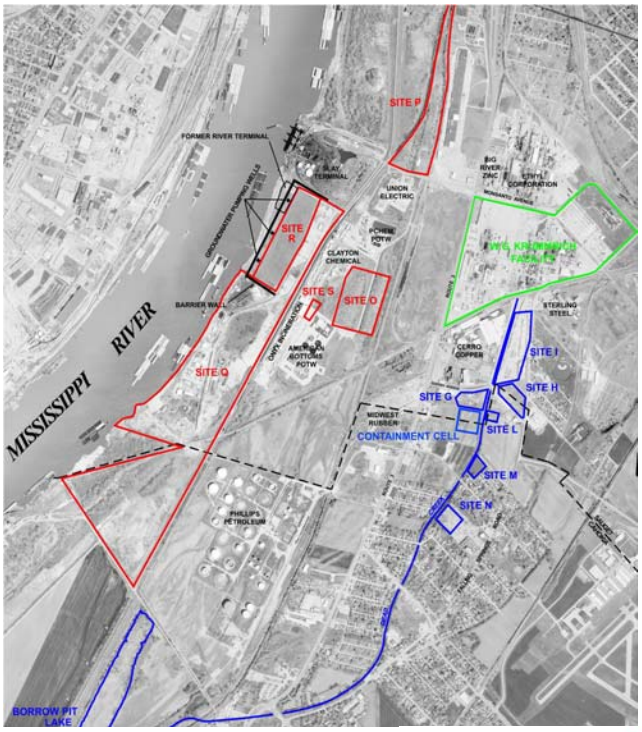
SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN

SOLUTIA INC.
W.G. KRUMMRICH FACILITY
SAUGET, ILLINOIS



Prepared by:
Solutia Inc.
575 Maryville Centre Dr.
St. Louis, Missouri 63141

July 2005

TABLE OF CONTENTS

FIGURES

TABLES

APPENDICES

1.0 INTRODUCTION

On May 3, 2000, USEPA executed a Resource Conservation and Recovery Act (RCRA) 3008(h) Administrative Order on Consent for Solutia Inc.'s W.G. Krummrich facility in Sauget, Illinois (**Figures 1.1 and 1.2**). Solutia Inc. signed the Administrative Order on Consent, Docket No. R8H-5-00-003, on May 26, 2000. Sections VI.1a, 1b, 2, 3 and 5, respectively, required Solutia to submit a Description of Current Conditions Report, investigate the nature and extent of any releases at or from the W.G. Krummrich facility, stabilize groundwater migration and show that any discharge of groundwater to surface water is either insignificant or currently acceptable, control completed pathway human exposures to contamination and propose final corrective measures for the site.

To fulfill the requirements of the AOC Solutia submitted a Description of Current Conditions Report, performed site investigations for air, soil, DNAPL and groundwater, completed Environmental Indicator Determinations for Migration of Contaminated Groundwater under Control (CA750) and Current Human Exposure Under Control (CA725) and submitted a Final Corrective Measures Study as summarized in the following table:

Summary of Work Performed to Fulfill the Requirements of the W.G. Krummrich RCRA AOC (Docket No. R8H-5-00-003)

• **Description of Current Conditions Report**	**August 1, 2000**
• **Sediment, Surface Water and Fish Tissue Sampling**	**October and November 2000**
• **Ecological Risk Assessment**	**June 1, 2001**
• **CA750 Migration of Contaminated Groundwater Under Control Environmental Indicator Determination**	**May 26, 2004**
• **CA725 Current Human Exposure Under Control Environmental Indicator Determination**	**May 26, 2004**
• **Air, Soil, DNAPL and Groundwater Investigation**	**2003 and 2004**
• **Corrective Measures Study**	**August 27, 2004**

In addition to these actions, Solutia implemented or planned a number of removal and remedial actions at Sauget Area 1, Sauget Area 2 and the W.G. Krummrich Facility prior to and after the May 26, 2000 RCRA AOC. A time line of the various removal actions and remedial actions and estimated expenditures for each action are given below:

Time Line of Sauget Area 1, Sauget Area 2 and W.G. Krummrich Removal/Remedial Actions and Estimated Expenditures

Sauget Area 1	**2001**	**Dead Creek Culvert Replacement Removal Action**	**$750,000**
	2002	**Dead Creek Time Critical Sediment Removal Action**	**12,300,000**
	2004	**Dead Creek Segment B, D and F Soil Removal Action Plan**	
Sauget Area 2	**1979**	**Site R Capping**	
	1985	**Site R Riverbank Stabilization**	**750,000**
	2003/4	**Groundwater Migration Control System**	**25,400,000**
W.G. Krummrich	**1987**	**Route 3 Drum Site Impermeable Cap**	
	2000	**Sewer System Improvements**	**17,100,000**
	2001	**Chlorobenzene Process Area Spill**	
	2003	**Plant Process Area Permeable Covers**	**310,000**
		Estimated Total Expenditure	**$56,610,000**

In comments provided by USEPA in May 2005 regarding the Corrective Measures Study, the Agency identified specific supplemental investigations necessary to further characterize potential source areas and associated risks. These areas include the Route 3 Drum Site, Lot F, Former Chlor-Alkali Production Area, North Central Plant Process Area, and the Former Coal Storage Area. Solutia is providing this

supplemental soil and groundwater sampling work plan, which describes the planned sampling at these select locations as directed by the Agency.

This Supplemental Soil and Groundwater Sampling Work Plan includes the following sections:

Section 1.0	**Introduction**
Section 2.0	**Supplemental Groundwater Investigation**
Section 3.0	**Supplemental Soil Investigation**
Section 4.0	**Data Management, Evaluation and Reporting**
Section 5.0	**Schedule**
Section 6.0	**References**

The sections describe the locations for sampling, sampling procedures, laboratory analytical program, data validation, and reporting. Figures and Tables follow the text. Field Standard Operating Procedures (SOPs) and laboratory SOPs are includes in Appendices A and B, respectively.

2.0 SUPPLEMENTAL GROUNDWATER INVESTIGATION

2.1 Objectives and Approach of the Supplemental Groundwater Investigation

The Route 3 Drum Site [Solid Waste Management Unit (SWMU) 27], located in the southwest corner of Lot F, was used as a drum disposal site before 1946. The Route 3 Drum Site was the subject of several interim actions from January 1985 to October 1987 consisting of excavation and off-site disposal, capping and fencing.

As directed by USEPA, Solutia plans additional characterization of groundwater quality at the Route 3 Drum Site to determine if the work performed to date, in addition to the groundwater collection provided by the Sauget Area 2 Groundwater Migration Control System, is sufficient to protect human health and the environment. The additional characterization includes redeveloping and sampling eight existing monitoring wells. The wells are shown on **Figure 2.1**.

2.2 Monitoring Well Redevelopment

Monitoring wells GM-8, GM-31A, GM-31B, GM-31C, GM-54A, GM-54B, GM-58A, and GM-59A will be redeveloped to remove any accumulated sediment using a conventional groundwater pump, air-lift system, or equivalent methods suitable for well development. Construction information for the monitoring wells is shown on **Table 2.1**. Before well redevelopment begins, the wells will be inspected for security, damage, and evidence of tampering. If damage or tampering is evident, the project manager will be contacted for guidance. Groundwater depth and total well depth (and the presence of any non-aqueous phase liquids (NAPL)) will be measured to the nearest 1/100 ft using an electronic interface probe and documented. Each monitoring well will be redeveloped until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings. In addition, the turbidity of the development water will be observed to see if suspended fines have been removed. The field parameters will be recorded on monitoring well sampling sheets during redevelopment. **SOP-1** described the well redevelopment procedures.

2.3 Groundwater Sampling Procedures

Groundwater level measurements and samples will be obtained from the eight monitoring wells listed above and illustrated on **Figure 2.1**. Groundwater level measurements will be obtained in accordance with **SOP-2**. Groundwater samples will be analyzed for SVOCs (Method 8270C) and PCBs (Method 8082).

Groundwater samples will be collected using low-flow methodologies including a flow-through cell. General procedures for low-flow sample collection are described below. Additional details are included in **SOP-3**. The groundwater sampling will proceed from the least impacted wells to the most impacted, based on available information. Equipment used for sampling that could contact groundwater will be properly decontaminated before each use. Field instruments will be calibrated prior to use in accordance with the manufacturer's specifications.

Clean plastic sheeting will be placed around the well and ambient volatile organic compound (VOC) background levels in the immediate vicinity of the well will be measured (i.e., using a photoionization detector (PID) or a flame ionization detector (FID)). Once the well cap is removed, VOCs will be

measured at the rim of the well and the readings recorded in the logbook or on the groundwater sampling form.

Immediately prior to sampling, groundwater depths (and the presence of any NAPL) will be measured to the nearest 1/100 ft using an electronic interface probe and documented. If NAPL is present, efforts will be made to collect water above or below the NAPL. The depth to the bottom of each well will be measured immediately after sampling, to minimize disturbing the water column.

The monitoring wells will be purged using a conventional groundwater pump, suitable for low flow applications (i.e., bladder pump [or equivalent]). Prior to purging, the pump will slowly be lowered to a depth in the well as described in the SOP. When purging first begins, the pump flow rate will be started at approximately 100mL/min or the lowest flow rate possible. Water level measurements and flow rate measurements will be taken every 2 minutes until they indicate that significant drawdown within the well is not occurring. Measurements will be scaled back to every five minutes when drawdown reaches equilibrium. Ideally, drawdown will be limited to 25% of the distance between the top of the screen and the pump intake. If significant drawdown occurs, the well will be pumped dry. After being pumped dry, the well will be periodically gauged until the water level has recharged to approximately 90% of the original, static level prior to sampling. If in 24 hours the well has not reached 90% static recovery, the well will be sampled.

Each monitoring well will be purged until pH, specific conductance, dissolved oxygen and oxygen reduction potential stabilize over a minimum of three successive flow-through cell volumes. In addition, temperature and turbidity will be measured but not used as sampling criteria. The field parameters will be measured and recorded on monitoring well sampling sheets during purging. The allowable ranges for the criteria used to determine stabilization is provided below:

- pH +/- 0.2 units
- Conductivity +/- 3%
- DO +/- 10% or +/-0.2 mg/L, whichever is greatest
- ORP +/- 20 mV

After the relevant parameters have stabilized, the flow-through cell will be bypassed for sampling. Groundwater samples will be collected at a flow rate not greater than 0.5 L/min (to minimize aeration) using the same pump used for purging. Personnel conducting the groundwater sampling will wear clean disposable protective gloves. The appropriate sample containers as described in **Table 2.2** will be filled in the order below:

- Semivolatile organic compounds
- Polychlorinated biphenyls

To verify field and laboratory procedures, quality assurance/quality control (QA/QC) samples consisting of duplicate samples, matrix spike/matrix spike duplicate (MS/MSD) samples and equipment blanks will be collected and submitted to the laboratory. QA/QC samples will be collected at a frequency of 10% for duplicates and blanks and 5% for MS/MSDs.

Equipment test checks are to be used to ensure QA/QC for the equipment used in field work. In order to reduce the potential for exposure to hazardous materials and limit the possibility of cross contamination of samples, field personnel and equipment will comply with decontamination and IDW procedures as described in the following section.

For proper identification in the field and proper tracking by the analytical laboratory, investigative and QC samples will be labeled in a clear and consistent fashion. Sample labels will be waterproof, or sample containers will be sealed in plastic bags.

A completed sample label will be attached to each investigative or QC sample. The following will be recorded with permanent ink on sample labels by the field sampling team:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

The sample identification system for groundwater sampling will involve the following nomenclature “AAA-BB” where:

“AAA” will denote the monitoring well

- GM8- Monitoring Well Location

“BB” will denote QA/QC sample

- EB- equipment blank
- AD- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate
- TB- Trip Blank

All samples will be placed on ice inside a cooler immediately following sampling. Sampling containers will be packed in such a way as to help prevent breakage and cross-contamination. Samples will be shipped in coolers, each containing a chain-of-custody form and ice packs to maintain inside temperature at approximately 4°C. Sample coolers will then be sealed between the lid and sides of the cooler with a custody seal prior to shipment. The custody seal will be an adhesive-backed tape that easily rips if it is disturbed. Samples will be shipped to the laboratory by common overnight carrier.

Sample transportation will comply with U.S. Department of Transportation and IICAO/IATA (1999)

regulations. Special sampling packing provisions will be made for samples requiring additional protection.

Field personnel will maintain a sampling log sheet containing information sufficient to allow reconstruction of the sample collection and handling procedures at a later time. Chain-of-custody procedures will be instituted and followed throughout the sampling activities. Samples are physical evidence and will be handled according to strict chain-of-custody protocols. The field sampler is personally responsible for the care and custody of the sample until transferred.

Field personnel will record the following information with permanent ink on the chain-of-custody:

- Project identification and number,
- Sample description/location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Number of sample containers,
- Analysis requested/comments,
- Sampler signature/date/time, and
- Air bill number.

A chain-of-custody document providing all information, signatures, dates, and other information, as required on the example chain-of-custody form included in **SOP-4** will be completed by the field sampler and provided for each sample cooler.

Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists of:

- Delivery of samples to the laboratory sample custodian, and
- Signature of the laboratory sample custodian on the chain-of-custody document as receiving the samples, and signature of sampler, as relinquishing the samples.

If a carrier is used to take samples between the sampler and the laboratory, a copy of the air bill must be attached to the chain-of-custody to maintain proof of custody.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. The field sampler will sign the chain-of-custody form when relinquishing custody, make a copy to keep with the field logbook, and include the original form in an air-tight plastic bag in the sample cooler with the associated samples.

The laboratory will assign a number for each sample upon receipt. That sample number will be placed on the sample label.

Additional information concerning sample handling, preservation and tracking is contained in **SOPs** 4 and

5.

2.4 Field Documentation

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, development sheets, sampling data sheets, and field logbooks. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler,

- Samples collected,
- Water level,
- Product level if necessary, and
- Depth to bottom of well.

Entries will be signed and dated, and any entry which is to be deleted will have a single cross out which is signed and dated.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Additional information concerning documentation is contained in **SOP-4**.

2.5 Decontamination and Investigation Derived Waste

During well redevelopment and sampling, field personnel and equipment will undergo decontamination procedures to ensure the health and safety of those present, to maintain sample integrity, and to minimize the movement of contamination between the work area and off-site locations. Equipment used on-site will be decontaminated prior to beginning work, between sampling locations and/or uses, and prior to demobilizing from the site. Non-disposable purging and sampling equipment will be decontaminated between each sample acquisition by washing with an Alconox® or equivalent detergent wash, a potable water rinse, and a distilled water rinse. Personnel and small equipment decontamination will be performed at the sample locations. Disposable sampling equipment, such as gloves will be collected and bagged on a daily basis and managed in accordance with Solutia procedures. Redevelopment water will be containerized and handled in accordance with Solutia Procedures. Refer to **SOP-6** for decontamination procedures.

3.0 SUPPLEMENTAL SOIL INVESTIGATION

3.1 Lot F

As requested by the USEPA, additional samples will be collected in the vicinity of sample locations S0205, S0206, and S0208 due to PCBs detected in these samples. During previous investigations, PCBs in surface soil [0-2 feet below ground surface (ft bgs)] were detected in Lot F at the above listed sample locations and in nearby soil at the Route 3 Drum Site during the 1986 cleanup. The PCB concentration in exposed surface soil at sample location S0205 was 2.5 mg/kg, which exceeds the TACO Tier 1 criteria for direct contact with soils of 1 mg/kg. A total of four surficial soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for PCBs including:

- One soil sample collected from a depth of 0 to 2 ft bgs located at the midpoint between soil sample locations S0205 and S0206 (new location S0214);
- One soil sample collected from a depth of 0 to 2 ft bgs located at the midpoint between soil sample locations S0205 and S0208; (new location S0215); and
- Two other soil samples collected from a depth of 0 to 2 ft bgs located 100 ft north and 100 ft south of soil sample location S0205 (new locations S0216 and S0217, respectively); .

The areal extent of SVOC and lead-containing fill will be assessed in the vicinity of sample location S0110. Sample location S0110 is located in a fill area as indicated from the S0110 boring log and a July 4, 1940 aerial photo. The sample collected from S0110 had detections of 13.2 mg/kg of total PAHs, 1 mg/kg benzo(a)pyrene, and 300 mg/kg lead in exposed surface soil (0-2 ft bgs). The detection for benzo(a)pyrene exceeded direct contact TACO Screening Criteria of 0.8 mg/kg, and the detection of lead approached the TACO Screening Criteria for direct contact. A total of six surficial soil samples will be collected from the locations shown in **Figure 3.1** including:

- Four soil samples collected and analyzed for SVOCs from a depth of 0 to 2 ft bgs located 100 ft north, south, east and west of soil sample location S0110 (new locations S0115-S0118, respectively); and
- Two soil samples collected and analyzed for SVOCs and lead using Method 8270C and 6010B respectively from a depth of 0 to 2 ft bgs 200 ft north and south of soil sample location S0110 (new locations S0119 and S0120, respectively).

The data will be used to help define this fill area and assess the potential human health and ecological risks.

The areal and vertical extent of VOC and SVOC-containing soils that exceed the TACO Tier I criteria for soil to groundwater leaching will be determined in the vicinity of sample location LF-4. According to USEPA, aerial photos indicate past activity (e.g., surface impoundment, disturbed ground) in the area of the LF-series soil sample locations. The LF-series soil sample locations at the southwest corner of Lot F were sampled at 18 to 20 ft bgs and found contain VOCs and SVOCs. LF-4 contained benzene, carbazole, nBnitrosodiphenylamine, and dichloromethane above the TACO Tier 1 soil to groundwater

leaching criteria. A total of four subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for VOCs and SVOCs including:

- Four soil samples collected and analyzed for VOCs and SVOCs from a depth of 18 to 20 ft bgs located 100 ft north, south, east and west of soil sample location LF-4 (new locations S0121, S0122, S0123 and S0124, respectively.

The samples to be collected are described in **Table 3.1**.

3.2 Former Chlor-Alkali Production Area

As requested by the USEPA, the areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria will be determined in the area of locations S0916, S0917, S0919, and S0920. A portion of the Former Chlor-Alkali Production Area including samples from soil borings S0916, S0917, S0919, and S0920 exceeds TACO Tier 1 criteria for direct contact with soils for Mercury. The areal extent of this contamination will be defined east of S0916 between S0922 and S0923; west and south of S0917 between S0911 and S0912, and S0910 and S0911; and northwest of S0919 between S0913 and S0914. The deepest sample at 7 to 10 ft bgs obtained at S0919, S0916, and S0920 exceeds the TACO Tier 1 criteria for direct contact with soils for Mercury. Mercury contamination is present in the fill, clayey silt, and silty clay but has not been defined in the deeper sand, which had not been encountered in the borings. A total of 15 subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for Mercury to define the areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria including:

- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0910 and S0911 (new location S0925);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0911 and S0912 (new location S0926);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0913 and S0914 (new location S0927);
- Three soil samples collected from depths of 2 to 3 ft, 6 to 7 ft and 9 to 10 ft bgs at the midpoint between soil sample locations S0922 and S0923 (new location S0928);
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0916;
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0919; and
- One soil sample collected from a depth of 13 to 15 ft bgs at soil sample location S0920.

Soils will be sampled and analyzed to confirm whether PCB concentrations are consistently less than the 25 ppm screening criterion in the area of sample locations S0904 and S0905. PCBs in the Former Chlor-Alkali Production Area were detected at 13 and 5 ppm at soil sample locations S0904 and S0905,

respectively within the fill between 9 and 13 ft bgs. A total of nine subsurface soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for PCBs including:

- On soil sample collected from 4 to 6 ft bgs at soil sample location S0902;
- On soil sample collected from 6 to 8 ft bgs at soil sample location S0902;
- On soil sample collected from 2 to 4 ft bgs at soil sample location S0903;
- On soil sample collected from 10 to 12 ft bgs at soil sample location S0907;
- On soil sample collected from 4 to 6 ft bgs at soil sample location S1003;
- On soil sample collected from 3 to 5 ft bgs at soil sample location S1004;
- On fill sample collected from the midpoint between soil sample locations S0904 and S0905 (new location S0929;
- On fill sample collected from the midpoint between soil sample locations S0904 and S0906 (new location S0930; and
- On fill sample collected from the midpoint between soil sample locations S0905 and S0907 (new location S0940).

The samples to be collected are described in **Table 3.1**.

3.3 North Central Plant Process Area

As requested by the USEPA, the presence of VOCs or SVOCs will be determined in the area of S0403. VOCs or SVOCs were not detected between 2 and 4 ft bgs for sample location S0403, although a strong odor and elevated PID reading were noted in the boring log. No intermediate or deep samples were collected from this sample location. Two soil samples will be collected at sampling depths where strong odors and elevated PID readings were noted in the boring log and analyzed for VOCs, SVOCs, Pesticides, Herbicides, and PCBs for sample location S0403 as shown in **Figure 3.1** including:

- One fill/soil sample collected from 1 to 3 ft bgs; and
- One fill/soil sample collected from 10 to 12 ft bgs.

The areal extent of VOCs that exceed either the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria will be assessed in the area of S0408 and S0409. Aerial photographs indicate that the area including sample locations S0408 and S0409 was a tank farm from at least 1940 through the 1980's. Soil sample locations S0408 and S0409 had elevated chlorobenzene, 1,3-dichloropropene, toluene, ethylbenzene, and xylene (VOC) concentrations at an intermediate depth. A total of four soil samples will be collected from the locations shown in **Figure 3.1** from the intermediate depth with the highest PID reading or most obviously impacted depth between ground surface and 15 ft bgs and analyzed for VOCs including:

- One soil sample collected 100 ft north of soil sample location S0408 (new location S0430);
- One soil sample collected 100 ft northeast of soil sample location S0408 (new location S0432);

- One soil sample collected 100 ft southwest of soil sample location S0408 (new location S0431); and
- One soil sample collected 100 ft west of soil sample location S0408 (new location S0433).

The samples to be collected are described in **Table 3.1**.

3.4 Former Coal Storage Area

As requested by the USEPA, soil samples will be collected to determine the potential risks associated with surface fill in the former coal storage area. The boring logs of soil sample locations S1101, S1102, and S1103 show that fill is present at all three sample locations, varying from 2 to 9 ft bgs. Surface soil samples were not obtained to determine the potential risks associated with surface fill. Therefore, a total of three soil samples will be collected from the locations shown in **Figure 3.1** and analyzed for SVOCs.

- One surface soil collected from 0 to 2 ft bgs at soil sample location S1101;
- One surface soil collected from 0 to 2 ft bgs at soil sample location S1102; and
- One surface soil collected from 0 to 2 ft bgs at soil sample location S1103.

The samples to be collected are described in **Table 3.1**.

3.5 Soil Analytical Parameters

Severn Trent Laboratories will analyze the soil samples for VOCs, SVOCs, Lead, Mercury, PCBs, Pesticides, and Herbicides as specified for each soil sample as described in the above subsections using the following methods:

Parameter	Analytical Method
VOCs	**USEPA Method 8260B**
SVOCs	**USEPA Method 8270C**
Lead	**USEPA Method 6010B**
Mercury	**USEPA Method 7471A**
PCBs	**USEPA Method 8082**
Pesticides	**USEPA Method 8081A**
Herbicides	**USEPA Method 8151A**

Chemical analysis will be conducted in accordance with the Laboratory SOPs located in **Appendix B. Table 3.2** describes the sample container size, preservation and hold times for soil.

3.6 Soil Sampling Procedures

Borings for soil sample collection will be advanced using direct push technology (Geoprobe®). The Geoprobe® hydraulically drives a stainless steel, acetate-lined MacroCore® sampler (2-inch diameter by 4-foot length) to the desired subsurface sample depths. Continuous samples will be collected from the surface to the proposed sampling depths. The subsurface stratigraphy will be logged during drilling operations by a qualified URS field scientist in accordance with the Unified Soil Classification System (USCS) protocols and **SOP-7**. The field scientist will note soil attributes such as color, particle size, consistency, moisture content, structure, plasticity, odor (if obvious) and organic content (if visible). Soil samples from each boring will be visually evaluated for evidence of impact and screened in the field using

a PID. Scaled, color digital photographs will be taken of selected soil samples to provide a record of materials present at this site. At the completion of each soil boring, the boreholes will be backfilled with bentonite chips, and the X-Y coordinates of each location will be obtained. Probing procedures are described in **SOP-8**. PID and sample screening procedures are included in **SOPs 9 and 10**. Samples for VOC analysis will be collected using low-level techniques (**SOP-11**).

To verify field and laboratory procedures, quality assurance/quality control (QA/QC) samples consisting of duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) samples may be collected and submitted to the laboratory. QA/QC sampling will be taken at a frequency of 10% for duplicates and blanks and 5% for MS/MSDs. Trip blanks will be included with each shipment of samples planned for VOC analysis.

Equipment test checks are to be used to ensure QA/QC for the equipment used in field work.

For proper identification in the field and proper tracking by the analytical laboratory, investigative and QC samples will be labeled in a clear and consistent fashion. Sample labels will be waterproof, or sample containers will be sealed in plastic bags.

A completed sample label will be attached to each investigative or QC sample. The following will be recorded with permanent ink on sample labels by the field sampling team:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location (if not already encoded in the sample number),
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

The sample identification system for soil will involve the following nomenclature “S-AA-BB-C-D-EEE” where:

“AA” will denote

- ## - Sample Area

“BB” will denote

- ##- Sample Number

“C” will denote

- #- Initial Sample Depth

“D” will denote

- #- Final Sample Depth

"EEE" will denote QA/QC sample

- DUP- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate

For example, S-04-18-1-2 indicates the soil sample was obtained from sample area 4, the North Production Area, for boring number 18, from 1 to 2 ft bgs.

Samples will be placed on ice inside a cooler immediately following sampling. Sampling containers will be packed in such a way as to help prevent breakage and cross-contamination. Samples will be shipped in coolers, each containing a chain-of-custody form and ice and ice packs to maintain inside temperature at approximately 4°C. Sample coolers will then be sealed between the lid and sides of the cooler with a custody seal prior to shipment. The custody seal will be an adhesive-backed tape that easily rips if it is disturbed. Samples will be shipped to the laboratory by common overnight carrier.

Sample transportation will comply with U.S. Department of Transportation and IICAO/IATA (1999) regulations. Special sampling packing provisions will be made for samples requiring additional protection.

Field personnel will maintain a sampling log sheet containing information sufficient to allow reconstruction of the sample collection and handling procedures at a later time. Chain-of-custody procedures will be instituted and followed throughout the sampling activities. Samples are physical evidence and will be handled according to strict chain-of-custody protocols. The field sampler is personally responsible for the care and custody of the sample until transferred.

Field personnel will record the following information with permanent ink on the chain-of-custody:

- Project identification and number,
- Sample description/location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Number of sample containers,
- Analysis requested/comments,
- Sampler signature/date/time, and
- Air bill number.

A chain-of-custody document providing all information, signatures, dates, and other information, as required on the example chain-of-custody form included in **SOP-4** will be completed by the field sampler and provided for each sample cooler.

Samples will remain in the custody of the sampler until transfer of custody is completed. Transfer consists

of:

- Delivery of samples to the laboratory sample custodian, and
- Signature of the laboratory sample custodian on the chain-of-custody document as receiving the samples, and signature of sampler, as relinquishing the samples.

If a carrier is used to take samples between the sampler and the laboratory, a copy of the air bill must be attached to the chain-of-custody to maintain proof of custody.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the chain-of-custody. The field sampler will sign the chain-of-custody form when relinquishing custody, make a copy to keep with the field logbook, and include the original form in an air-tight plastic bag in the sample cooler with the associated samples.

The laboratory will assign a number for each sample upon receipt. That sample number will be placed on the sample label.

Additional information concerning sample handling, preservation and tracking is contained in **SOPs 4 and 5**.

3.7 Field Documentation

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, boring log, and soil sampling data sheets and field logbook. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler, and
- Samples collected.

Entries will be signed and dated, and any entry which is to be deleted will have a single cross out which is signed and dated. Photographic records will be developed through the use of digital photographs, showing pre-sampling and post-sampling conditions at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Additional information concerning documentation is contained in **SOP-4**.

3.8 Decontamination and Investigation Derived Waste

The field activities (i.e., sampling, and IDW handling) and data management methods and procedures will follow those discussed in Sections 2.0 and 3.0. Field personnel and equipment will undergo decontamination procedures as described above in Section 2.6. Decontamination fluids will be containerized and handled as discussed in Section 2.6. The IDW including PPE, will be handled as discussed in Section 2.6. Soil cuttings derived from the soil borings will be containerized in 55-gallon, open-top steel drums and managed in accordance with Solutia procedures. The drums will be identified by marking reference information on the lid (e.g., boring number, drum contents, date filled, etc.). Refer to **SOP-6** for decontamination procedures.

4.0 DATA MANAGEMENT, EVALUATION AND REPORTING

4.1 Data Management

The field data and documentation will become a part of the final file. The final file will be the central repository for all documents, which constitute evidence relevant to sampling and analysis activities as described in this plan. URS is the custodian of the file and maintains the contents of files for the site, including all relevant records, logs, field logbooks, pictures, subcontractor reports, data reviews, and the database management system.

The following documentation will supplement the chain-of-custody records:

- Field logbooks and data
- Field collection report
- Photographs and drawings
- Contractor and subcontractor reports
- Correspondence.

The file must be maintained in a secured, limited access area until all submittals for the project have been reviewed and approved, and for a minimum of six years past the submittal date of the final report.

Upon completion of the analyses, URS will begin assimilating the field and laboratory notes. In this way, the file for the samples will be generated. The final file for the samples will be stored at URS and will consist of the following:

- Laboratory data packages, including summary and raw data from the analysis of environmental and QC samples, chromatograms, mass spectra, calibration data, work sheets, and sample preparation notebooks
- Chain-of-custody records
- Data validation reports.

Analytical data will be provided in hard copy and electronic formats. Electronic data will be loaded into a database to facilitate data evaluation and reporting. The data presented in the report will include the data flags provided by STL as well as the qualifiers assigned by URS.

4.2 Data Quality Objectives

The general objective of quality assurance is to collect defensible environmental data of known quality that is adequate for the data's intended use. To accomplish this, data quality objectives (DQOs) were developed. DQOs are qualitative and quantitative statements which clarify the study objective, define the most appropriate types of data to collect, determine the most appropriate conditions from which to collect data, and specify acceptable decisions regarding the data's usage (including reporting limits) to ensure that the collected data will fulfill the project objectives.

Detected analyte concentrations will be compared to relevant Illinois Environmental Protection Agency (IEPA) Tiered Approach to Corrective Action Objectives guidelines. **Tables 4.1** and **4.2** lists the DQOs for

constituents. URS will work with STL to attain the lowest reporting limits to meet the project objectives. However due to technical constraints, achieving reporting limits that are lower than the DQOs might not be feasible for all compounds.

4.3 Quality Assurance Objectives for Measurement

Quality assurance objectives for measurement data are usually expressed in terms of precision, accuracy, completeness, representativeness, and comparability. The investigation will not be considered invalid if these criteria are not fully achieved but variances will trigger QA/QC measures to evaluate, and correct if necessary, any problem areas. The control limits for precision and accuracy as well as detection limits for each laboratory analysis are listed in **Tables 4.3** and **4.4**.

Precision is a measure of the degree to which two or more measurements are in agreement. Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per ten analytical samples. Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) for two or more replicate samples. The equations to be used for precision in this plan are presented in the model QAPP Section 14.2. The precision objective for laboratory analysis will be ± 25 percent RPD for groundwater and ± 50 percent RPD for soil between field duplicates.

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation, and holding times. Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or laboratory control samples (LCSs), and the determination of percent recoveries. The equation to be used for accuracy in this plan is presented in the model QAPP Section 14.1.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness for this project will be at least 90 percent. Laboratory completeness is a measure of the amount of valid measurements obtained from all the laboratory measurements taken in the project. The equation for completeness is presented in the model QAPP Section 14.3. Laboratory completeness for this project will be at least 95 percent. Results assigned an “R” qualifier would be unusable.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the plan is followed and that proper sampling techniques are used. Representativeness in the laboratory data is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing the field duplicate samples and matrix spike duplicate samples. The sampling network is designed to provide data representative of site conditions. During development of this network, consideration is given to existing analytical data, past site practices, and physical setting and processes.

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability can be related to precision and accuracy since these quantities are measures of data

reliability. Samples from the same media are considered comparable if the procedures for collecting the samples are complied with and if the units of measurement are the same. Comparability is assured through the use of a laboratory for this project that uses established and approved analytical methods, protocols, and a laboratory quality control program designed to establish consistency in the performance of the analytical process. All data will be subjected to strict QA/QC procedures and reported in a consistent manner to allow for comparison across data sets.

4.4 Data Validation

Data validation will be performed by the URS QA Manager in accordance with QA/QC criteria established in the USEPA Region 5 Model QAPP. Excursions from QA/QC criteria will be qualified based on guidance provided in the following documents where applicable to the reference methods:

- USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review. USEPA 540/R-94/012 (USEPA, 2001)
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. USEPA 540/R-94/013 (USEPA, 2004)

A Level III validation will be performed for all data, and a Level IV validation will be performed for approximately 10 percent of the data. Data validators will recalculate approximately 10 percent of the laboratory sample calculations using raw data when verifying sample results. In addition, data validators will review approximately 10 percent of the raw data to verify that compound identification was performed correctly and transcription errors are not present.

Data quality will be evaluated using laboratory or method control limits. Any control limits outside of the acceptable range shall be identified and reported. Sample data will be qualified based on excursions from laboratory or method control limits. Then, data validators will check corrective actions and results of reanalysis and document these events in the validation report.

Minor deficiencies in the data generation process noted in the data validation will result in approximation of sample data. Approximation of a data point indicates uncertainty in the reported concentration of the chemical but not its assigned identity. Major deficiencies noted in the data, validation will result in the rejection of sample results. Rejected data would be considered unusable for quantitative or qualitative purposes. Data qualifiers may include the following:

U Indicates that the compound was analyzed for, but was not detected. The sample quantitation limit is presented and adjusted for dilution and percent moisture. This qualifier is also used to signify that the detection limit of an analyte was raised as a result of analytes detected in laboratory and/or field blank samples.

J Indicates that the detected sample result should be considered approximate based on excursions from QA/QC criteria.

UJ Indicates that the detection limit for the analyte in this sample should be considered approximate based on excursions from QA/QC criteria.

R Indicates that the previously reported detection limit or sample result has been rejected due to a

major excursion from QA/QC criteria, for example percent recoveries of less than ten percent. The data should not be used for qualitative or quantitative purposes.

The following method specific QA/QC parameters will be evaluated (at a minimum) during the data validation, where applicable.

Analyses for VOCs and SVOCs (where applicable)

Holding times, sample preservation, and percent solids

Dilutions

GC/MS instrument performance (Level IV validation only)

Initial and continuing calibration (Level IV validation only)

Blank analysis

Surrogate recovery

MS/MSD analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Internal standards performance

Compound identification and quantification (Level IV validation only)

Documentation completeness

Overall assessment.

Analyses for Pesticides and PCBs, (where applicable):

Holding times, sample preservation, and percent solids

Dilutions

Initial and continuing calibration (Level IV validation only)

Blank analysis

Surrogate recovery

MS/MSD analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Compound identification and quantification (Level IV validation only)

Documentation completeness

Overall assessment.

Analyses for Herbicides, (where applicable):

- Holding times, sample preservation, and percent solids
- Dilutions
- Initial and continuing calibration (Level IV validation only)
- Blank analysis
- Surrogate recovery
- MS/MSD analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Compound identification and quantification (Level IV validation only)
- Documentation completeness
- Overall assessment.

Analyses for Metals, (where applicable):

- Holding times, sample preservation, and percent solids
- Dilutions
- Calibration (Level IV validation only)
- Blank analysis
- Spike analysis
- Field duplicate analysis
- Laboratory Control Sample (LCS) analysis
- Sample Result Verification (Level IV validation only)
- ICP Interference Check Samples (Level IV validation only)
- Laboratory Duplicates
- ICP Serial Dilution
- Documentation completeness
- Overall assessment.

Analyses for Mercury, (where applicable):

- Holding times and sample preservation
- Dilutions
- Initial and continuing calibration (Level IV validation only)

Compound identification and quantification

Blank analysis

Spike analysis

Field duplicate analysis

Laboratory Control Sample (LCS) analysis

Laboratory Duplicates

Documentation completeness

Overall assessment.

The data will be provided to URS in an electronic format that will be uploaded into a database to facilitate data review and evaluation. The report tables will be generated from the database. These tables will include the data flags provided by Savannah Labs as well as the data validation qualifiers assigned by URS.

Laboratory provided results (Form 1's) will also be included in the final report with the transcribed data qualifiers.

4.5 Reporting

The Supplemental Soil and Groundwater Sampling Report will be prepared and submitted to USEPA after sampling, sample analysis and data validation are completed. The report will include a groundwater-elevation contour map, a summary of the laboratory analytical data (soil and groundwater), and a discussion of the following objectives requested by the USEPA in a letter dated May 4, 2005.

The report will provide the following types of information for the Lot F Drum: the general procedures for and frequency of inspections and maintenance of the cap, a list of activities and costs of inspection and maintenance included in the final corrective measures array analysis and a figure delineating the boundaries of the Route 3 Drum Site and location of each monitoring well sampled with individual constituent concentrations found in groundwater at each monitoring well sampled. The results of this work will be used to evaluate if the interim actions conducted at the Lot F drum Site in addition to groundwater collection at the Sauget Area 2 Groundwater Migration Control System, is sufficient to protect human health and the environment.

The report will define the vertical and areal extent of contamination at Lot F, the Former Chlor-Alkali Production Area, the North Central Plant Process Area, and the Former Coal Storage Area and address the following specific requests.

Lot F:

- The areal extent of PCB-containing soils and the associated human health and ecologic risk,
- the extent of the fill area,
- and the areal and vertical extent of VOC and SVOC-containing soils that exceed the TACO Tier I criteria for soil to groundwater leaching in Lot F.

Former Chlor-Alkali Production Area:

- The areal and vertical extent of soils containing mercury at concentrations higher than the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria,
- and a confirmation that PCB concentrations are consistently less than the 25 ppm screening criteria.

North Central Process Area:

- The results of the two soil samples collected for sample location S0403 to determine if the elevated PID readings indicated the presence of VOCs,
- and the areal and vertical extent of VOCs that exceed either the TACO Tier I criteria for direct contact with soils or the soil to groundwater leaching criteria.

Former Coal Storage Area:

- The results and analysis of three soil samples collected to determine the potential risks of the contaminants present in the fill.

5.0 SCHEDULE

The proposed schedule for the supplemental soil and groundwater sampling work to be completed is as follows:

Task	July	August	September
Work Plan Approved			
Field Sampling Activities			
Laboratory Analysis			
Data Review and Validation			
Report Compilation			

6.0 REFERENCES

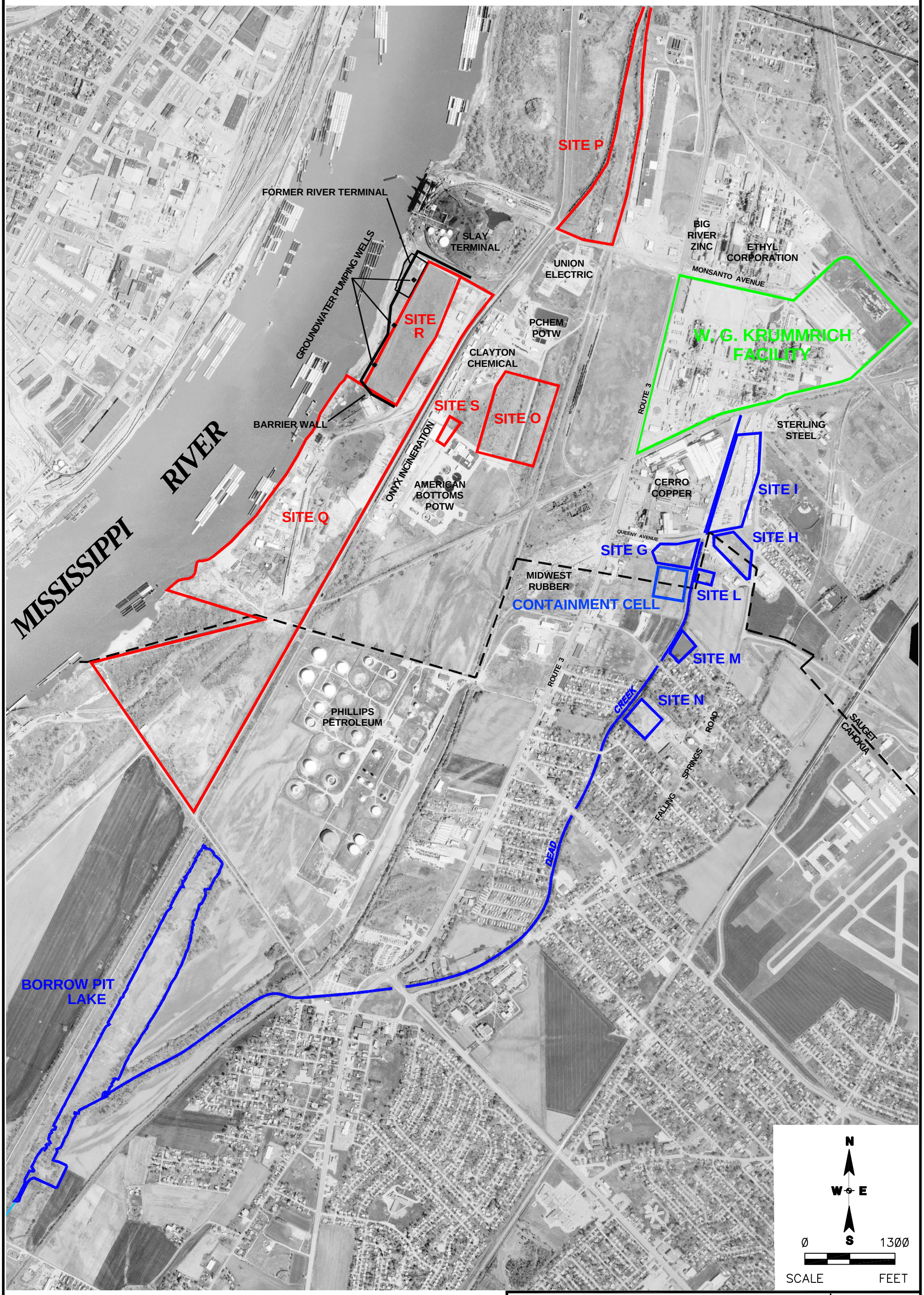
U.S. Environmental Protection Agency (USEPA) Region 5, 1998, *RCRA QAPP Instructions*, April 1998, Chicago, IL.

URS Corporation, 2004. *RCRA Corrective Measures Study (CMS), Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois*.

URS Corporation, 2005. *RCRA Corrective Measures Study (CMS), Response to Comments, Solutia Inc., W.G. Krummrich Facility, Sauget, Illinois*.

URS Corporation, 2005. *RCRA Corrective Measures Study, Response to May 4, 2005 USEPA Comments, Solutia Inc.., W.G. Krummrich Facility Investigation, Sauget, Illinois*.

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING\FIGURES\FIG-1.1 SITE LOCATION MAP.DWG Last edited: JUN. 23, 05 @ 12:46 p.m. by: djdeguil0



LEGEND

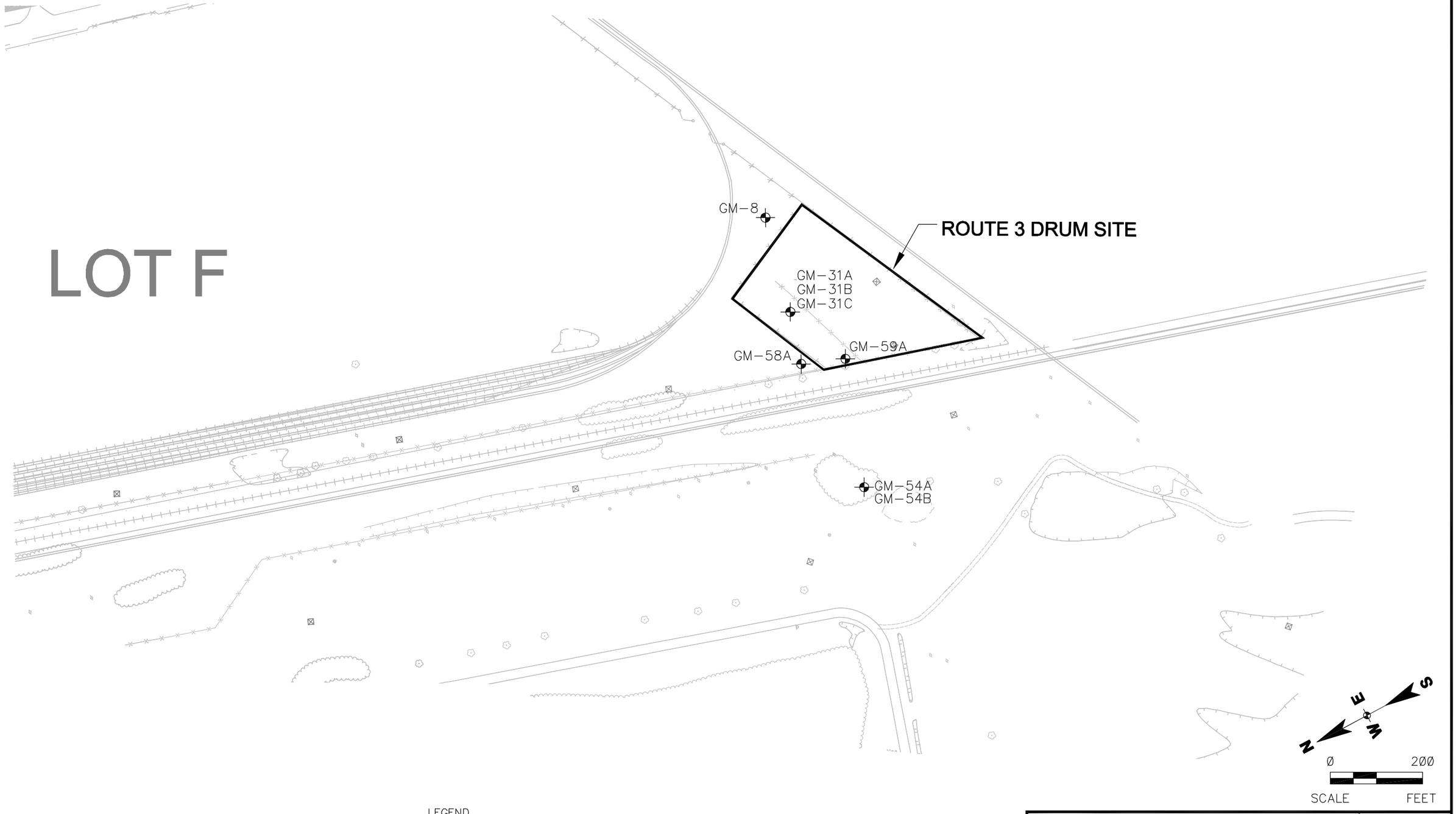
- W.G. KRUMMRICH FACILITY
- SAUGET AREA #1
- SAUGET AREA #2

SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000
URS	
DRN. BY: 2/9/05 DSGN. BY: tja CHKD. BY:	Site Location Map
	FIG. NO. 1.1

SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS	PROJECT NO. 21561573.00000

URS

DRN. BY: 4/9/05 DSGN. BY: cm CHKD. BY:	Facility Location Map	FIG. NO. 1.2



LEGEND

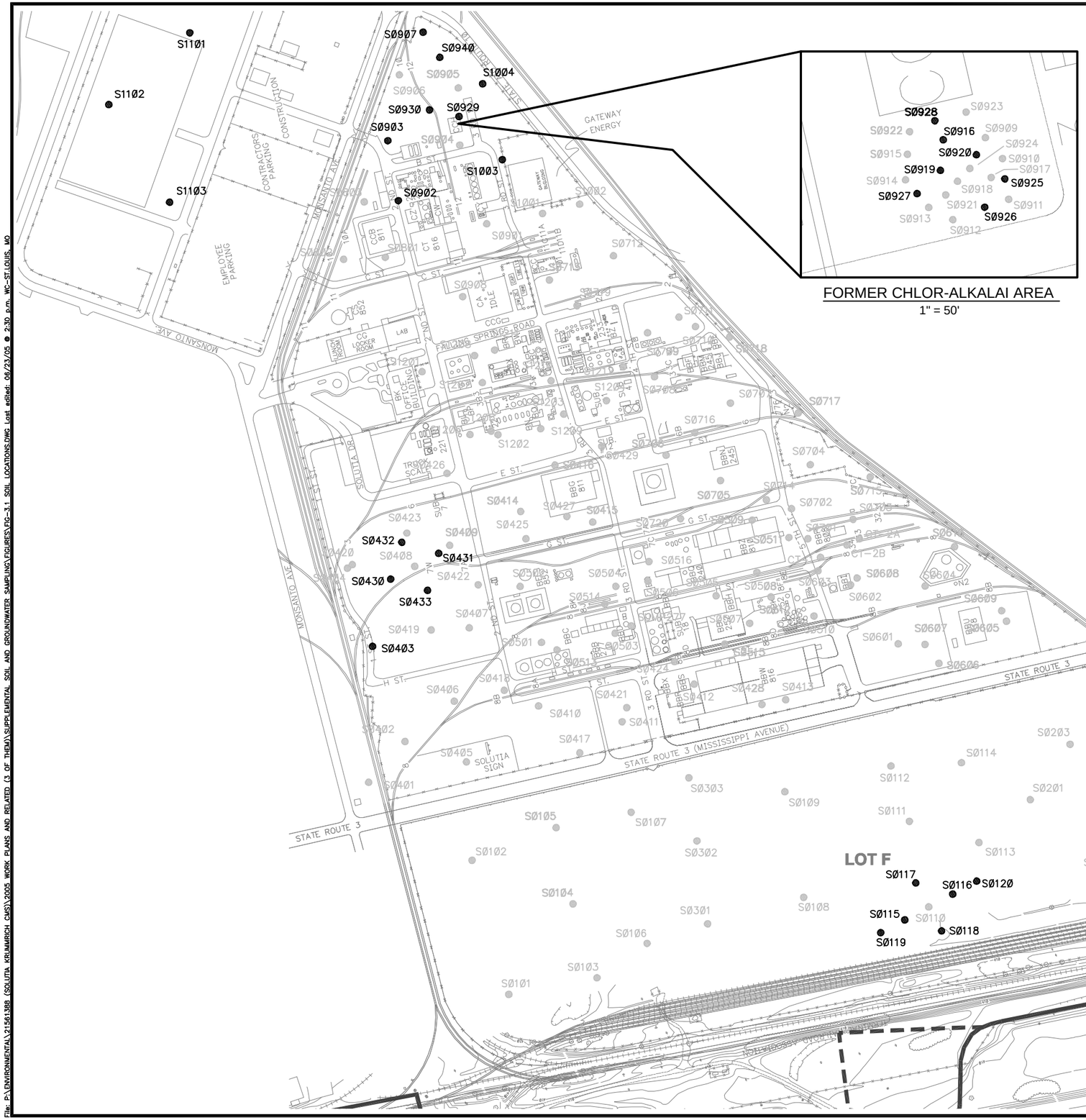
MONITORING WELL LOCATION

SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING PLAN W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS		PROJECT NO. 21561573.00000
URS		
DRN. BY:djd 2/10/05 DSGN. BY: bh CHKD. BY:	Monitoring Well Locations	FIG. NO. 2.1

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH CMS)\2005 WORK PLANS AND RELATED (3 OF THEM)\SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING\FIGURES\FIG-2.1 MW LOCATIONS.DWG Last edited: 06/23/05 @ 4:17 p.m. WC-ST.LOUIS, MO

LEGEND

● BORING LOCATION



FORMER CHLOR-ALKALAI AREA

1" = 50'

SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING W.G. KRUMMRICH FACILITY SAUGET, ILLINOIS		PROJECT NO. 21561573.00000
URS		
DRN. BY: 6/17/05 DSGN. BY: cm CHKD. BY:	Soil Sample Locations	FIG. NO. 3.1

File: P:\ENVIRONMENTAL\21561388 (SOLUTIA KRUMMRICH GWSI)\2005 WORK PLANS AND RELATED\3 OF THEM\SUPPLEMENTAL SOIL AND GROUNDWATER SAMPLING\FIGURES\FIG-3-1 SOIL LOCATIONS.DWG Last edited: 06/23/05 @ 2:30 p.m. WC-ST.LOUIS, MO

TABLE 2.1
MONITORING WELL COMPLETION SUMMARY
ROUTE 3 DRUM SITE

Monitoring Well Identification	Top of Casing Elevation (ft MSL)	Total Well Depth (ft btoc)	Bottom of Well Elevation (ft MSL)	Screened Interval (ft btoc)	Screened Interval Elevation (ft MSL)
GM-31A	418.63	33.15	385.48	(19.00-39.00)	(399.63-379.63)
GM-31B	418.92	86.21	332.71	(65.50-85.50)	(353.42-333.42)
GM-31C	419.29	119.4	299.89	(97.00-117.00)	(322.29-302.29)
GM-54A	404.93	40.75	364.18	(66.50-86.50)	(338.43-318.43)
GM-54B	405.11	92.15	312.96	(66.50-86.50)	(338.61-318.61)
GM-58A	414.24	41.8	372.44	(19.40-39.40)	(394.84-374.84)
GM-59A	413.57	41.53	372.04	(19.00-39.00)	(394.57-374.57)
GM-8	418.49	35.76	382.73	(19.00-34.00)	(399.49-384.49)

Notes:
1) MSL=Mean Sea Level
2) btoc= below top of casing
3) The total well depths for GM-54A, GM-54B, and GM-58A shown on the table were measured between January 24, 2000 and February 1, 2000.
4) The total well depths for GM-31A, GM-31B, GM-31C, GM-59A, and GM-8 shown on the table were measured June 15, 2005.

Table 2.2 Recommended Sample Containers, Preservation, and Hold Times for Parameters Measured in Wastewater and Groundwater Matrices

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Base/Neutrals/Acids	8270	2 x 1-L Amb G	None (6)	7 days/40 days

PCBs by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Polychlorinated Byphenols	8082	1-L Amb G	None (6)	7 days/40 days

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0214	Lot F	Midpoint between S0205 and S0206	0-2	PCBs	USEPA Method 680
S0215	Lot F	Midpoint between S0205 and S0208	0-2	PCBs	USEPA Method 680
S0216	Lot F	100' north of S0205	0-2	PCBs	USEPA Method 680
S0217	Lot F	100' south of S0205	0-2	PCBs	USEPA Method 680
S0115	Lot F	100' north of S0110	0-2	SVOCs	USEPA Method 8270C
S0116	Lot F	100' south of S0110	0-2	SVOCs	USEPA Method 8270C
S0117	Lot F	100' east of S0110	0-2	SVOCs	USEPA Method 8270C
S0118	Lot F	100' west of S0110	0-2	SVOCs	USEPA Method 8270C
S0119	Lot F	200' north of S0110	0-2	SVOCs Lead	USEPA Method 8270C USEPA Method 6010B
S0120	Lot F	200' south of S0110	0-2	SVOCs Lead	USEPA Method 8270C USEPA Method 6010B
S0121	Lot F	100' north of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0122	Lot F	100' south of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0123	Lot F	100' east of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0124	Lot F	100' west of LF-4	18-20	VOCs SVOCs	USEPA Method 8260B USEPA Method 8270C
S0925	Former Chlor-Alkali Production Area	Midpoint between S0910 and S0911	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0926	Former Chlor-Alkali Production Area	Midpoint between S0911 and S0912	2-3 6-7 9-10	Mercury	USEPA Method 7470C

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0927	Former Chlor-Alkali Production Area	Midpoint between S0913 and S0914	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0928	Former Chlor-Alkali Production Area	Midpoint between S0922 and S0923	2-3 6-7 9-10	Mercury	USEPA Method 7470C
S0916	Former Chlor-Alkali Production Area	S0916	13-15	Mercury	USEPA Method 7470C
S0919	Former Chlor-Alkali Production Area	S0919	13-15	Mercury	USEPA Method 7470C
S0920	Former Chlor-Alkali Production Area	S0920	13-15	Mercury	USEPA Method 7470C
S0902	Former Chlor-Alkali Production Area	S0902	4-6 6-8	PCBs	USEPA Method 680
S0903	Former Chlor-Alkali Production Area	S0903	2-4	PCBs	USEPA Method 680
S0907	Former Chlor-Alkali Production Area	S0907	10-12	PCBs	USEPA Method 680
S1003	Former Chlor-Alkali Production Area	S1003	4-6	PCBs	USEPA Method 680
S1004	Former Chlor-Alkali Production Area	S1004	3-5	PCBs	USEPA Method 680
S0929	Former Chlor-Alkali Production Area	Midpoint between S0904 and S0905	Fill sample	PCBs	USEPA Method 680
S0930	Former Chlor-Alkali Production Area	Midpoint between S0904 and S0906	Fill sample	PCBs	USEPA Method 680

Table 3.1
Soil Sample Description

Sample Location ID	Area	Location Description	Sample Depth (ft bgs)	Sample Parameters	EPA Method
S0940	Former Chlor-Alkali Production Area	Midpoint between S0905 and S0907	Fill sample	PCBs	USEPA Method 680
S0403	North Central Plant Process Area	S0403	1-3 10-12	VOCs SVOCs Pesticides Herbicides PCBs	USEPA Method 8260B USEPA Method 8270C USEPA Method 8081A USEPA Method 8151A USEPA Method 680
S0430	North Central Plant Process Area	100' north of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0431	North Central Plant Process Area	100' south of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0432	North Central Plant Process Area	100' east of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S0433	North Central Plant Process Area	100' west of S0408	Highest PID Reading 0-15	VOCs	USEPA Method 8260B
S1101	Former Coal Storage Area	S1101	0-2	SVOCs	USEPA Method 8270C
S1102	Former Coal Storage Area	S1102	0-2	SVOCs	USEPA Method 8270C
S1103	Former Coal Storage Area	S1103	0-2	SVOCs	USEPA Method 8270C

Table 3.2 Recommended Sample Containers, Preservation, and Hold Time for Parameters Measured in Soil and Solid Matrices

General Chemistry

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Organic Carbon, Total (TOC)	Lloyd Kahn (Combustion)	250-ml P	None	28 days

Volatile Organics – GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Volatiles	8260(5035)	3 x 5-g Encore plus125-ml G Amb	5ml 5% sodium bisulfate solution, methanol, or frozen in water	14 days

Semivolatiles by GC/MS

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Base/Neutrals/Acids	8270	250-ml or 500-ml amb G	None	14 days/40 days

Organochloride Pesticides and PCBs by GC

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Polychlorinated Byphenols	8082	500-ml amb G	None	14 days/40 days

Chlorinated Herbicides

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Herbicides	8151A	500-ml amb G	None (6)	14 days/40 days

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Metals (except Mercury)	ICP: 6010 GFAA:7000-series	250 ml P	None	6 months

Metals

PARAMETER	METHOD REFERENCE	ROUTINE CONTAINER	CHEMICAL PRESERVATIVE	HOLD TIME
Mercury	7471A	250 ml P or G	None	28 days

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Polychlorinated Biphenyls as Aroclors by GC/EC							
PCB 1016 (MS)	12674-11-2	8082	0.14	1.0	NA	Not Available	NA
PCB 1221	11104-28-2	8082	0.50	2.0	NA	Not Available	NA
PCB 1232	11141-16-5	8082	0.22	1.0	NA	Not Available	NA
PCB 1242	53469-21-9	8082	0.22	1.0	NA	Not Available	NA
PCB 1248	12672-29-6	8082	0.33	1.0	NA	Not Available	NA
PCB 1254	11097-69-1	8082	0.20	1.0	NA	Not Available	NA
PCB 1260 (MS)	11096-82-5	8082	0.17	1.0	NA	Not Available	NA
PCB 1268	11100-14-4	8082	0.10	1.0	NA	Not Available	NA
Surrogates		8082					
Decachlorobiphenyl	2051-24-3	8082	NA	NA	NA	Not Available	NA
2,4,5,6-Tetrachloro-m-xylene	877-09-8	8082	NA	NA	NA	Not Available	NA
Semivolatiles in Groundwater by GC/MS							
1,2,4,5-Tetrachlorobenzene	95-94-3	8270 (3520)	1.2	10.00	NA	Not Available	NA
1,2,4-Trichlorobenzene (MS)	120-82-1	8270 (3520)	0.54	10.00	70.00	IEPA TACO Class I	Yes
1,2-Dichlorobenzene	95-50-1	8270 (3520)	1.0	10.00	600.00	IEPA TACO Class I	Yes
1,2-Diphenyl hydrazine	122-66-7	8270 (3520)	0.97	10.00	NA	Not Available	NA
1,3,5-Trinitrobenzene	99-35-4	8270 (3520)	0.62	10.00	210.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1,3-Dichlorobenzene	541-73-1	8270 (3520)	0.55	10.00	5.50	Region 9 (Tap Water)	No
1,4-Dichlorobenzene(MS)	106-46-7	8270 (3520)	0.52	10.00	75.00	IEPA TACO Class I	Yes
1,4-Dioxane	123-91-1	8270 (3520)	2.3	10.00	1.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
1,4-Naphthoquinone	130-15-4	8270 (3520)	0.67	10.00	NA	Not Available	NA
1-Methylnaphthalene	90-12-0	8270 (3520)	0.62	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
1-Naphthylamine	134-32-7	8270 (3520)	1.2	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
2,3,4,6-Tetrachlorophenol	58-90-2	8270 (3520)	0.47	10.00	NA	Not Available	NA
2,4,5-Trichlorophenol	95-95-4	8270 (3520)	0.63	10.00	700.00	IEPA TACO Class I	Yes
2,4,6-Trichlorophenol	88-06-2	8270 (3520)	0.70	10.00	10.00	IEPA TACO Class I	Yes
2,4-Dichlorophenol	120-83-2	8270 (3520)	0.66	10.00	21.00	IEPA TACO Class I	Yes
2,4-Dimethylphenol	105-67-9	8270 (3520)	1.0	10.00	140.00	IEPA TACO Class I	Yes
2,4-Dinitrophenol	51-28-5	8270 (3520)	5.0	50.00	14.00	IEPA TACO Class I	No
2,4-Dinitrotoluene (MS)	121-14-2	8270 (3520)	0.56	10.00	0.02	IEPA TACO Class I	No
2,6-Dichlorophenol	87-65-0	8270 (3520)	0.69	10.00	NA	Not Available	NA
2,6-Dinitrotoluene	606-20-2	8270 (3520)	0.57	10.00	0.31	IEPA TACO Class I	No
2-Acetylaminofluorene	53-96-3	8270 (3520)	1.4	10.00	NA	Not Available	NA
2-Chloronaphthalene	91-58-7	8270 (3520)	0.62	10.00	NA	Not Available	NA
2-Chlorophenol (MS)	95-57-8	8270 (3520)	0.72	10.00	35.00	IEPA TACO Class I	Yes
2-Methyl phenol	95-48-7	8270 (3520)	0.70	10.00	350.00	IEPA TACO Class I	Yes
2-Methylnaphthalene	91-57-6	8270 (3520)	0.53	10.00	140.00	IEPA TACO Class I - Surrogate Chemical: Napthalene (per USEPA National Center for Environmental Assessment)	Yes
2-Naphthylamine	91-59-8	8270 (3520)	0.64	10.00	NA	Not Available	NA
2-Nitroaniline	88-74-4	8270 (3520)	0.69	50.00	1.00	Region 9 (Tap Water)	No
2-Nitrophenol	88-75-5	8270 (3520)	0.73	10.00	NA	Not Available	NA
2-Picoline	109-06-8	8270 (3520)	0.87	10.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	No
3- and 4-Methyl phenol	106-44-5	8270 (3520)	0.66	10.00	35.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - April 2, 2003	Yes
3,3'-Dichlorobenzidine	91-94-1	8270 (3520)	1.0	20.00	20.00	IEPA TACO Class I	Yes
3,3'-Dimethylbenzidine	119-93-7	8270 (3520)	2.5	20.00	NA	Not Available	NA
3-Methylcholanthrene	56-49-5	8270 (3520)	1.4	10.00	NA	Not Available	NA
3-Nitroaniline	99-09-2	8270 (3520)	0.69	50.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
4,6-Dinitro-2-methylphenol	534-52-1	8270 (3520)	1.0	50.00	0.01	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
4-Aminobiphenyl	92-67-1	8270 (3520)	0.68	10.00	NA	Not Available	NA
4-Bromophenyl phenyl ether	101-55-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
4-Chloro-3-methyl-phenol (MS)	59-50-7	8270 (3520)	0.55	10.00	NA	Not Available	NA
4-Chloroaniline	106-47-8	8270 (3520)	0.52	20.00	28.00	IEPA TACO Class I	Yes
4-Chlorophenylphenyl ether	7005-72-3	8270 (3520)	0.56	10.00	NA	Not Available	NA
4-Nitroaniline	100-01-6	8270 (3520)	0.85	50.00	NA	Not Available	NA
4-Nitrophenol (MS)	100-02-7	8270 (3520)	3.4	50.00	290.00	Region 6 (Tap Water)	Yes
4-Nitroquinoline-1-oxide	56-57-5	8270 (3520)	5.0	20.00	NA	Not Available	NA
5-Nitro-o-toluidine	99-55-8	8270 (3520)	1.2	10.00	NA	Not Available	NA
7,12-Dimethylbenz(a)anthracene	57-97-6	8270 (3520)	1.3	10.00	NA	Not Available	NA
a,a-Dimethylphenethylamine	122-09-8	8270 (3520)	10	2000.00	NA	Not Available	NA
Acenaphthene (MS)	83-32-9	8270 (3520)	0.64	10.00	420.00	IEPA TACO Class I	Yes
Acenaphthylene	208-96-8	8270 (3520)	0.58	10.00	NA	Not Available	NA
Acetophenone	98-86-2	8270 (3520)	0.83	10.00	NA	Not Available	NA
Aniline	62-53-3	8270 (3520)	0.65	20.00	23.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Anthracene	120-12-7	8270 (3520)	0.88	10.00	2100.00	IEPA TACO Class I	Yes
Aramite	140-57-8	8270 (3520)	1.7	10.00	NA	Not Available	NA
Benzidine	92-87-5	8270 (3520)	14	80.00	NA	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Benzo(a)anthracene	56-55-3	8270 (3520)	0.58	10.00	0.13	IEPA TACO Class I	No
Benzo(a)pyrene	50-32-8	8270 (3520)	1.0	10.00	0.20	IEPA TACO Class I	No
Benzo(b)fluoranthene	205-99-2	8270 (3520)	0.78	10.00	0.18	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Benzo(g,h,i)perylene	191-24-2	8270 (3520)	1.0	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Benzo(k)fluoranthene	207-08-9	8270 (3520)	0.73	10.00	0.17	IEPA TACO Class I	No
Benzoic acid	65-85-0	8270 (3520)	2.5	50.00	28000.00	IEPA TACO Class I	Yes
Benzyl alcohol	100-51-6	8270 (3520)	0.80	10.00	2100.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Bis(2-chloroethoxy) methane	111-91-1	8270 (3520)	0.78	10.00	NA	Not Available	NA
Bis(2-chloroethyl) ether	111-44-4	8270 (3520)	0.72	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	111-44-4	8270 (3520)	0.82	10.00	10.00	IEPA TACO Class I	Yes
Bis(2-ethylhexyl) phthalate	117-81-7	8270 (3520)	0.98	10.00	6.00	IEPA TACO Class I	No
Butyl benzyl phthalate	85-68-7	8270 (3520)	0.70	10.00	1400.00	IEPA TACO Class I	Yes
Carbazole	86-74-8	8270 (3520)	0.82	10.00	3.40	Region 9 (Tap Water)	No
Chrysene	218-01-9	8270 (3520)	1.0	10.00	1.50	IEPA TACO Class I	No
Diallate	2303-16-4	8270 (3520)	1.6	10.00	NA	Not Available	NA
Dibenz(a,h)anthracene	53-70-3	8270 (3520)	1.0	10.00	0.30	IEPA TACO Class I	No
Dibenzofuran	132-64-9	8270 (3520)	0.69	10.00	28.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Diethyl phthalate	84-66-2	8270 (3520)	0.59	10.00	5600.00	IEPA TACO Class I	Yes
Dimethoate	60-51-5	8270 (3520)	0.64	10.00	NA	Not Available	NA
Dimethylphthalate	131-11-3	8270 (3520)	0.59	10.00	NA	Not Available	NA
Di-n-butyl phthalate	84-74-2	8270 (3520)	0.82	10.00	4000.00	IEPA TACO Class I	Yes
Di-n-octyl phthalate	117-84-0	8270 (3520)	0.80	10.00	140.00	IEPA TACO Class I	Yes
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	88-85-7	8270 (3520)	2.5	10.00	7.00	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Diphenylamine/ Nitrosodiphenylamine	122-39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Disulfoton	298-04-4	8270 (3520)	0.78	10.00	0.28	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Ethyl methanesulfonate	62-50-0	8270 (3520)	0.90	10.00	NA	Not Available	NA
Ethyl parathion	56-38-2	8270 (3520)	0.89	10.00	168.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Famphur	52-85-7	8270 (3520)	2.2	10.00	NA	Not Available	NA
Fluoranthene	206-44-0	8270 (3520)	1.0	10.00	280.00	IEPA TACO Class I	Yes
Fluorene	86-73-7	8270 (3520)	0.55	10.00	280.00	IEPA TACO Class I	Yes
Hexachlorobenzene	118-74-1	8270 (3520)	1.1	10.00	0.06	IEPA TACO Class I	No
Hexachlorobutadiene	87-68-3	8270 (3520)	1.0	10.00	1.40	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Hexachlorocyclopentadiene	77-47-4	8270 (3520)	5.0	10.00	50.00	IEPA TACO Class I	Yes
Hexachloroethane	67-72-1	8270 (3520)	1.0	10.00	7.00	IEPA TACO Class I	No
Hexachlorophene	70-30-4	8270 (3520)	47	5000.00	NA	Not Available	NA
Hexachloropropene	1888-71-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Indeno(1,2,3-cd)pyrene	193-39-5	8270 (3520)	0.62	10.00	0.43	IEPA TACO Class I	No
Isophorone	78-59-1	8270 (3520)	0.67	10.00	1400.00	IEPA TACO Class I	Yes
Isosafrole	120-58-1	8270 (3520)	0.96	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
m-Dinitrobenzene	99-65-0	8270 (3520)	0.68	10.00	0.70	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Methapyrilene	91-80-5	8270 (3520)	1.9	2000.00	NA	Not Available	NA
Methyl parathion	298-00-0	8270 (3520)	0.78	10.00	NA	Not Available	NA
Methylmethanesulfonate	66-27-3	8270 (3520)	0.85	10.00	NA	Not Available	NA
Naphthalene	91-20-3	8270 (3520)	0.76	10.00	140.00	IEPA TACO Class I	Yes
Nitrobenzene	98-95-3	8270 (3520)	0.93	10.00	3.50	IEPA TACO Class I	No
Nitrosodiphenylamine/ Diphenylamine	86-30-6, 122 39-4	8270 (3520)	0.80	10.00	175.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
N-Nitrosodiethylamine	55-18-5	8270 (3520)	0.82	10.00	NA	Not Available	NA
N-Nitrosodimethylamine	62-75-9	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosodi-n-butylamine	924-16-3	8270 (3520)	0.78	10.00	NA	Not Available	NA
N-Nitrosodi-n-propylamine(MS)	621-64-7	8270 (3520)	0.62	10.00	1.80	IEPA TACO Class I	No
N-Nitrosomethylethylamine	10595-95-6	8270 (3520)	2.6	10.00	NA	Not Available	NA
N-Nitrosomorpholine	59-89-2	8270 (3520)	1.1	10.00	NA	Not Available	NA
N-Nitrosopiperidine	100-75-4	8270 (3520)	1.2	10.00	NA	Not Available	NA
N-Nitrosopyrrolidine	930-55-2	8270 (3520)	0.57	10.00	NA	Not Available	NA
o,o,o-Triethyl-phosphorothioate	126-68-1	8270 (3520)	1.3	10.00	NA	Not Available	NA
o-Toluidine	95-53-4	8270 (3520)	0.58	10.00	NA	Not Available	NA
p-(Dimethylamino)azobenzene	60-11-7	8270 (3520)	1.2	10.00	NA	Not Available	NA
Pentachlorobenzene	608-93-5	8270 (3520)	1.0	10.00	NA	Not Available	NA
Pentachloronitrobenzene	82-68-8	8270 (3520)	1.2	10.00	21.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
Pentachlorophenol (MS)	87-86-5	8270 (3520)	2.5	50.00	1.00	IEPA TACO Class I	No
Phenacetin	62-44-2	8270 (3520)	0.82	10.00	NA	Not Available	NA

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Phenanthrene	85-01-8	8270 (3520)	0.74	10.00	0.21	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Phenol (MS)	108-95-2	8270 (3520)	0.99	10.00	100.00	IEPA TACO Class I	Yes
Phorate	298-02-2	8270 (3520)	0.83	10.00	14.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
p-Phenylenediamine	106-50-3	8270 (3520)	37	2000.00	NA	Not Available	NA
Pronamide	23950-58-5	8270 (3520)	0.72	10.00	NA	Not Available	NA
Pyrene(MS)	129-00-0	8270 (3520)	1.0	10.00	210.00	IEPA TACO Class I	Yes
Pyridine	110-86-1	8270 (3520)	0.81	50.00	7.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	No
Safrole	94-59-7	8270 (3520)	0.99	10.00	NA	Not Available	NA
Sulfotepp	3689-24-5	8270 (3520)	0.70	10.00	NA	Not Available	NA
Thionazin	297-97-2	8270 (3520)	0.99	10.00	NA	Not Available	NA
Surrogates							
2-Fluorobiphenyl	321-60-8	8270 (3520)	NA	NA	NA	Not Available	NA
2-Fluorophenol	367-12-4	8270 (3520)	NA	NA	NA	Not Available	NA
Nitrobenzene-d5	98-95-3	8270 (3520)	NA	NA	3.50	IEPA TACO Class I	No
Phenol-d5	108-95-2	8270 (3520)	NA	NA	100.00	IEPA TACO Class I	No
Terphenyl-d14	26140-60-3	8270 (3520)	NA	NA	NA	Not Available	NA
2,4,6-Tribromophenol		8270 (3520)	NA	NA	NA	Not Available	NA
Non-Routine Analytes							
Atrazine	1912-24-9	8270 (3520)	0.89	10.00	3.00	IEPA TACO Class I	No

Table 4.1
Data Quality Objective Analysis for Groundwater

PARAMETER	CAS NUMBER	METHOD	MDL (ug/L)	RL (ug/L)	Class I Screening Value (ug/L)	Ref Description	Screening Value > RL?
Benzaldehyde	100-52-7	8270 (3520)	1.0	10.00	700.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,1-Biphenyl (1,1-Diphenyl)		8270 (3520)	0.68	10.00	NA	Not Available	NA
Caprolactam	105-60-2	8270 (3520)	0.84	10.00	3500.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
2,3-Dimethylphenol	526-75-0	8270 (3520)	0.86	10.00	NA	Not Available	NA
2,5-Dimethylphenol	95-87-4	8270 (3520)	0.93	10.00	NA	Not Available	NA
2,6-Dimethylphenol	576-26-1	8270 (3520)	0.69	10.00	NA	Not Available	NA
3,4-Dimethylphenol	95-65-8	8270 (3520)	0.79	10.00	NA	Not Available	NA
2,5-Dinitrophenol	329-71-5	8270 (3520)	0.99	50.00	NA	Not Available	NA
3-Nitrophenol	554-84-7	8270 (3520)	0.78	10.00	NA	Not Available	NA
Phenyl ether (Diphenyl oxide)	101-84-8	8270 (3520)	1.0	10.00	NA	Not Available	NA
1,2,3,5-Tetrachlorobenzene	634-90-2	8270 (3520)	0.75	10.00	NA	Not Available	NA
1,2,3-Trichlorobenzene	87-61-6	8270 (3520)	0.59	10.00	70.00	IEPA Chemicals not in TACO Tier 1 Tables - IEPA Toxicity Assesment Unit - October 1, 2004	Yes
1,3,5-Trichlorobenzene	108-70-3	8270 (3520)	0.62	10.00	NA	Not Available	NA

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Soil to GW Screening Value (mg/L)	Ingestion Industrial-Commercial (mg/kg)	Inhalation Industrial-Commercial (mg/kg)	Ingestion Construction Worker (mg/kg)	Inhalation Construction Worker (mg/kg)	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Metals Parameters												
Aluminum	6010 (3050)	4.5	2.0	3.50	1,000,000	--	200,000	--	200,000.00	IEPA TACO - Class I	N.C.	No
Antimony	6010 (3050)	0.45	2.0	0.006	820	--	82	--	82.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Arsenic	6010 (3050)	0.67	1.0	0.05	--	1,200	61	25,000	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Barium	6010 (3050)	0.30	1.0	2.00	140,000	910,000	14,000	870,000	14,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Beryllium	6010 (3050)	0.017	0.40	0.004	4,100	21,000	410	44,000	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Boron	6010 (3050)	1.30	5.0	2.00	180,000	1,000,000	18,000	1,000,000	18,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Cadmium	6010 (3050)	0.22	0.50	0.005	2,000	2,800	200	59,000	200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Calcium	6010 (3050)	2.4	50	Not Available								
Chromium	6010 (3050)	0.13	1.0	0.10	6,100	420	4,100	690	420.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Cobalt	6010 (3050)	0.17	1.0	1.00	120,000	--	12,000	--	12,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Copper	6010 (3050)	0.17	2.0	0.65	82,000	--	8,200	--	8,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Iron	6010 (3050)	4.2	5.0	5.00	--	--	--	--	0.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Lead	6010 (3050)	0.21	0.50	0.0075	400	--	400	--	400.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Magnesium	6010 (3050)	1.2	50	Not Available								
Manganese	6010 (3050)	0.21	1.0	0.15	96,000	91,000	9,600	8,700	8,700.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Molybdenum	6010 (3050)	0.33	1.0	Not Available								
Nickel	6010 (3050)	0.26	4.0	0.10	41,000	21,000	4,100	440,000	4,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Potassium	6010 (3050)	1.3	100	Not Available								
Selenium	6010 (3050)	0.90	2.5	0.05	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Silver	6010 (3050)	0.099	1.0	0.05	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Sodium	6010 (3050)	50	100	Not Available								
Strontium	6010 (3050)	0.15	1.0	4.20	1,000,000	--	120,000	--	120,000.00	IEPA TACO - Class I	N.C.	Yes

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Soil to GW Screening Value (mg/L)	Ingestion Industrial-Commercial (mg/kg)	Inhalation Industrial-Commercial (mg/kg)	Ingestion Construction Worker (mg/kg)	Inhalation Construction Worker (mg/kg)	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Thallium	6010 (3050)	1.30	2.5	0.002	160	--	160	--	160.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Tin	6010 (3050)	4.0	10	4.20	1,000,000	--	120,000	--	120,000.00	IEPA TACO - Class I	N.C.	No
Titanium	6010 (3050)	0.040	1.0	Not Available								
Vanadium	6010 (3050)	0.14	1.0	0.049	14,000	--	1,400	--	1,400.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Zinc	6010 (3050)	0.75	2.0	5.00	610,000	--	61,000	--	61,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No
Mercury	7471	0.0040	0.020	0.002	610	540,000	61	52,000	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	N.C.	No

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
General Chemistry Parameters												
Ammonia (as N)	350.1(EPA-CE:3-140)	0.075	0.15	Not Available								
Polychlorinated Biphenyls as Aroclors by GC/EC (Sonication Extraction)												
PCB-1016	8082 (3550)	6.7	33	Not Available								
PCB 1221	8082 (3550)	6.8	67	Not Available								
PCB 1232	8082 (3550)	6.2	33	Not Available								
PCB-1242	8082 (3550)	7.5	33	Not Available								
PCB-1248	8082 (3550)	8.0	33	Not Available								
PCB-1254	8082 (3550)	5.2	33	Not Available								
PCB-1260	8082 (3550)	6.4	33	Not Available								
PCB-1268	8082 (3550)	11	33	Not Available								
Surrogates												
2,4,5,6-Tetrachloro-m-xylene	8082 (3550)	NA	NA	Not Available								
Decachlorobiphenyl	8082 (3550)	NA	NA	Not Available								
Chlorinated Pesticides by GC/EC (Sonication Extraction)												
4,4'-DDD	8081 (3550)	0.30	3.3	Not Available								
4,4'-DDE	8081 (3550)	0.30	3.3	Not Available								
4,4'-DDT (MS)	8081 (3550)	0.27	3.3	Not Available								
Aldrin (MS)	8081 (3550)	0.14	1.7	0.5	0.3	6.6	6.1	9.3	0.30	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
alpha Chlordane	8081 (3550)	0.17	1.7	Not Available								
alpha-BHC	8081 (3550)	0.52	1.7	Not Available								
beta-BHC	8081 (3550)	0.47	1.7	Not Available								
Chlorobenzilate	8081 (3550)	3.8	17	Not Available								
delta-BHC	8081 (3550)	0.23	1.7	Not Available								
Dieldrin (MS)	8081 (3550)	0.35	3.3	0.004	0.4	2.2	7.8	3.1	0.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Endosulfan I	8081 (3550)	0.16	1.7	18	12,000	--	1,200	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Endosulfan II	8081 (3550)	0.27	3.3	18	12,000	--	1,200	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Endosulfan sulfate	8081 (3550)	0.37	3.3	Not Available								
Endrin (MS)	8081 (3550)	0.32	3.3	1	610	--	61	--	61.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Endrin aldehyde	8081 (3550)	0.65	3.3	Not Available								
Endrin ketone	8081 (3550)	0.32	3.3	Not Available								
gamma Chlordane	8081 (3550)	0.22	1.7	Not Available								
gamma-BHC (Lindane) (MS)	8081 (3550)	0.14	1.7	Not Available								
Heptachlor (MS)	8081 (3550)	0.32	1.7	23	1	11	28	16	1.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Heptachlor epoxide	8081 (3550)	0.21	1.7	0.7	0.6	9.2	2.7	13	0.60	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isodrin	8081 (3550)	0.33	3.3	Not Available								
Kepone	8081 (3550)	6.7	170	Not Available								
Methoxychlor	8081 (3550)	0.47	17	160	10,000	--	1,000	--	1,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Technical Chlordane	8081 (3550)	3	17	Not Available								
Toxaphene	8081 (3550)	12	170	31	5.2	170	110	240	5.20	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Surrogates												
Tetrachloro-m-xylene	8081 (3550)	NA	NA	Not Available								
Decachlorobiphenyl	8081 (3550)	NA	NA	Not Available								
Non-routine Compounds												
2,4'-DDD	8081 (3550)	3.3	3.3	Not Available								
2,4'-DDE	8081 (3550)	3.3	3.3	Not Available								
2,4'-DDT	8081 (3550)	3.3	3.3	Not Available								
Chlorinated Herbicides by GC/EC												
2,4-D (MS)	8151	0.97	8.3	1.5	20,000	--	2,000	--	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-DB	8151	1.0	8.3	Not Available								
2,4,5-T (MS)	8151	0.56	8.3	Not Available								
2,4,5-TP (Silvex) (MS)	8151	0.52	8.3	11	16,000	--	1,600	--	1,600.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dalapon	8151	10	330	0.85	61,000	--	6,100	--	6,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dicamba	8151	1.5	8.3	2.8	61,000	--	6,100	--	6,100.00	IEPA TACO - Class I	No	No
Dichlorprop	8151	1.8	8.3	Not Available								

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Dinoseb	8151	14	100	0.34	2,000	--	200	--	200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
MCPA	8151	42	2000		1,000	--	100	--	100.00	IEPA TACO - Class I	No	No
MCPP	8151	220	2000		2,000	--	2,000	--	2,000.00	IEPA TACO - Class I	No	No
Pentachlorophenol	8151	0.98	8.3	0.03	24	--	520	--	24.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Surrogates												
2,4-Dichlorophenyl acetic acid	8151	NA	NA	Not Available								
Non-Routine Compounds												
Picloram	8151	2.6	8.3	2	140,000	--	14,000	--	14,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Volatiles by GC/MS												
1,1,1,2-Tetrachloroethane	8260 (5035)	0.45	5.0	2	61,000	1,000	6,100	1,000	1,000.00	IEPA TACO - Class I	No	No
1,1,1-Trichloroethane	8260 (5035)	0.50	5.0	2	--	1,200	--	1,200	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,1,2,2-Tetrachloroethane	8260 (5035)	0.60	5.0	3.3	120,000	2,000	12,000	2,000	2,000.00	IEPA TACO - Class I	No	No
1,1,2-Trichloro-1,2,2-trifluoroethane	8260 (5035)	1.2	5.0	Not Available								
1,1,2-Trichloroethane	8260 (5035)	0.54	5.0	0.02	8,200	1,800	8,200	1,800	1,800.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,1-Dichloroethane	8260 (5035)	0.4	5.0	23	200,000	1,700	200,000	130	130.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,1-Dichloroethene (MS)	8260 (5035)	0.56	5.0	Not Available								
1,1-Dichloropropene	8260 (5035)	0.64	5.0	Not Available								
1,2,3-Trichlorobenzene	8260 (5035)	0.38	5.0	5.7	20,000	--	2,000	--	2,000.00	IEPA TACO - Class I	Yes	No
1,2,3-Trichloropropane	8260 (5035)	0.87	5.0	0.0001	0.82	1,000	18	1,000	0.82	IEPA TACO - Class I	No	No
1,2,4-Trichlorobenzene	8260 (5035)	0.46	5.0	5	20,000	3,200	2,000	920	920.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,2,4-Trimethylbenzene	8260 (5035)	0.4	5.0	18	100,000	110	10,000	0.73	0.73	IEPA TACO - Class I	Yes	No
1,2-Dibromo-3-chloropropane (DBCP)	8260 (5035)	1.0	10	0.002	4	17	89	0.11	0.11	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dibromoethane (EDB)	8260 (5035)	0.73	5.0	0.0004	0.07	0.32	1.5	0.45	0.07	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dichlorobenzene	8260 (5035)	0.53	5.0	17	180,000	560	18,000	310	310.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
1,2-Dichloroethane	8260 (5035)	0.59	5.0	0.02	63	0.7	1,400	0.99	0.70	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,2-Dichloroethenes (total)	8260 (5035)	0.62	10	Not Available								
1,2-Dichloropropane	8260 (5035)	0.42	5.0	0.03	84	23	1,800	0.5	0.50	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
1,3,5-Trimethylbenzene	8260 (5035)	0.34	5.0	10	100,000	71	10,000	0.46	0.46	IEPA TACO - Class I	Yes	No
1,3-Dichlorobenzene	8260 (5035)	0.5	5.0	0.2	1,800	570	180	570	180.00	IEPA TACO - Class I	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
1,3-Dichloropropane	8260 (5035)	0.56	5.0	Not Available								
1,4-Dichlorobenzene	8260 (5035)	0.47	5.0	2	--	17,000	--	340	340.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,2-Dichloropropane	8260 (5035)	0.59	5.0	Not Available								
2-Butanone (Methyl Ethyl Ketone-MEK)	8260 (5035)	1.8	25	17	1,000,000	25,000	120,000	710	710.00	IEPA TACO - Class I	No	No
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5035)	2.1	5.0	Not Available								
2-Chloroethyl vinyl ether	8260 (5035)	50.0	50	Not Available								
2-Chlorotoluene	8260 (5035)	0.5	5.0	Not Available								
2-Hexanone	8260 (5035)	2.0	25	1.3	82,000	110	8,200	0.72	0.72	IEPA TACO - Class I	No	No
3-Chloropropene (Allyl chloride)	8260 (5035)	2.3	5.0	Not Available								
4-Chlorotoluene	8260 (5035)	0.57	5.0	Not Available								
4-Methyl-2-pentanone (MIBK)	8260 (5035)	1.9	25	Not Available								
Acetone	8260 (5035)	2.6	50	16	200,000	100,000	200,000	100,000	100,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Acetonitrile	8260 (5035)	53	200	Not Available								
Acrolein	8260 (5035)	23	100	0.014	1,000	0.26	100	0.0017	0.0017	IEPA TACO - Class I	No	No
Acrylonitrile	8260 (5035)	16	100	0.0006	11	0.54	230	0.17	0.17	IEPA TACO - Class I	No	No
Benzene (MS)	8260 (5035)	0.52	5.0	0.03	100	1.6	2,300	2.2	1.60	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromobenzene	8260 (5035)	0.4	5.0	Not Available								
Bromochloromethane	8260 (5035)	0.47	5.0	Not Available								
Bromodichloromethane	8260 (5035)	0.42	5.0	0.6	92	3,000	2,000	3,000	92.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromoform	8260 (5035)	0.62	5.0	0.8	720	100	16,000	140	100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bromomethane	8260 (5035)	4.3	5.0	0.2	2,900	15	1,000	3.9	3.90	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Carbon disulfide	8260 (5035)	0.4	5.0	32	200,000	720	20,000	9	9.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Carbon tetrachloride	8260 (5035)	0.36	5.0	0.07	44	0.64	410	0.9	0.64	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chlorobenzene (MS)	8260 (5035)	0.84	5.0	1	41,000	210	4,100	1.3	1.30	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chloroethane	8260 (5035)	1.0	5.0	15	820,000	1,500	82,000	94	94.00	IEPA TACO - Class I	Yes	No
Chloroform	8260 (5035)	0.36	5.0	0.6	940	0.54	2,000	0.76	0.54	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chloromethane	8260 (5035)	0.42	5.0	0.14	8,200	170	820	1.1	1.10	IEPA TACO - Class I	No	No
cis-1,2-Dichloroethene	8260 (5035)	0.30	5.0	Not Available								
cis-1,3-Dichloropropene	8260 (5035)	0.45	5.0	0.004	57	2.1	1,200	0.39	0.39	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Dibromochloromethane	8260 (5035)	0.5	5.0	0.4	41,000	1,300	41,000	1,300	1,300.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dibromomethane	8260 (5035)	0.42	5.0	Not Available								
Dichlorodifluoromethane	8260 (5035)	0.90	5.0	48	410,000	390	180,000	26	26.00	IEPA TACO - Class I	Yes	No
Ethyl methacrylate	8260 (5035)	1.2	5.0	Not Available								
Ethylbenzene	8260 (5035)	0.6	5.0	13	200,000	400	20,000	58	58.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachlorobutadiene	8260 (5035)	0.68	5.0	Not Available								
Iodomethane	8260 (5035)	1.3	5.0	Not Available								
Isobutyl alcohol	8260 (5035)	83	200	Not Available								
Isopropylbenzene	8260 (5035)	0.35	5.0	86	200,000	760	20,000	4.9	4.90	IEPA TACO - Class I	Yes	No
m&p-Xylene	8260 (5035)	1.5	5.0	200	1,000,000	420	410,000	420	420.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Methacrylonitrile	8260 (5035)	31	100	Not Available								
Methyl methacrylate	8260 (5035)	2.0	5.0	Not Available								
Methyl t-butyl ether (MTBE)	8260 (5035)	0.69	50	Not Available								
Methylene chloride	8260 (5035)	0.87	5.0	0.02	760	24	12,000	34	24.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Naphthalene	8260 (5035)	1.8	5.0	12	41,000	270	4,100	1.8	1.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
n-Butylbenzene	8260 (5035)	0.42	5.0	11	20,000	53	2,000	53	53.00	IEPA TACO - Class I	Yes	No
n-Propylbenzene	8260 (5035)	0.47	5.0	2.6	20,000	260	2,000	260	260.00	IEPA TACO - Class I	No	No
o-Xylene	8260 (5035)	0.44	5.0	190	1,000,000	410	410,000	410	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Pentachloroethane	8260 (5035)	1.4	25	Not Available								
p-Isopropyltoluene	8260 (5035)	0.39	5.0	Not Available								
Propionitrile (ethylcyanide)	8260 (5035)	30	100	Not Available								
sec-Butylbenzene	8260 (5035)	0.45	5.0	15	20,000	42	2,000	42	42.00	IEPA TACO - Class I	Yes	No
Styrene	8260 (5035)	0.53	5.0	4	410,000	1,500	41,000	430	430.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
tert-Butylbenzene	8260 (5035)	0.52	5.0	6.5	20,000	77	2,000	77	77.00	IEPA TACO - Class I	Yes	No
Tetrachloroethene	8260 (5035)	0.91	5.0	Not Available								
Toluene (MS)	8260 (5035)	0.69	5.0	12	410,000	650	410,000	42	42.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
trans-1,2-Dichloroethene	8260 (5035)	0.37	5.0	Not Available								
trans-1,3-Dichloropropene	8260 (5035)	0.45	5.0	0.004	57	2.1	1,200	0.39	0.39	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
trans-1,4-Dichloro-2-butene	8260 (5035)	3.6	10	Not Available								
Trichloroethene (MS)	8260 (5035)	4.4	5.0	Not Available								
Trichlorofluoromethane	8260 (5035)	0.4	5.0	33	610,000	1,300	410,000	85	85.00	IEPA TACO - Class I	Yes	No
Vinyl acetate	8260 (5035)	0.83	10	170	1,000,000	1,600	200,000	10	10.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Vinyl chloride	8260 (5035)	0.59	5.0	0.01	7.9	1.1	170	1.1	1.10	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Xylenes (total)	8260 (5035)	1.9	10	150	1,000,000	320	410,000	320	320.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Surrogates												
p-Bromofluorobenzene	8260 (5035)	NA	NA	Not Available								
Dibromofluoromethane	8260 (5035)	NA	NA	Not Available								
Toluene-d8	8260 (5035)	NA	NA	Not Available								
Non-Routine Compounds												
1-Chlorohexane	8260 (5035)	0.72	5.0	Not Available								
Cyclohexane	8260 (5035)	10	10	Not Available								
Cyclohexanone	8260 (5035)	15	50	150	1,000,000	660	1,000,000	660	660.00	IEPA TACO - Class I	Yes	No
Diethyl ether	8260 (5035)	0.78	10	Not Available								
Furan	8260 (5035)	0.36	5.0	Not Available								
Methyl acetate	8260 (5035)	1.6	10	7	1,000,000	27,000	1,000,000	27,000	27,000.00	IEPA TACO - Class I	No	No
Methyl cyclohexane	8260 (5035)	0.48	10	Not Available								
Tetrahydrofuran	8260 (5035)	0.36	10	Not Available								
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)												
1,2,4,5-Tetrachlorobenzene	8270 (3550)	31	330	Not Available								
1,2,4-Trichlorobenzene (MS)	8270 (3550)	21	330	Not Available								
1,2-Dichlorobenzene	8270 (3550)	23	330	Not Available								
1,2-Diphenyl hydrazine	8270 (3550)	22	330	Not Available								
1,3,5-Trinitrobenzene	8270 (3550)	82	330	0.97	61,000	--	6,100	--	6,100.00	IEPA TACO - Class I	No	No
1,3-Dichlorobenzene	8270 (3550)	26	330	Not Available								
1,4-Dichlorobenzene (MS)	8270 (3550)	22	330	Not Available								
1,4-Dioxane	8270 (3550)	66	330	0.031	520	100,000	11,000	98,000	520.00	IEPA TACO - Class I	No	No
1,4-naphthoquinone	8270 (3550)	39	330	Not Available								
1-Methylnaphthalene	8270 (3550)	29	330	7.2	8,200	990	820	990	820.00	IEPA TACO - Class I	No	No
1-Naphthylamine	8270 (3550)	82	330	Not Available								
2,4,5-Trichlorophenol	8270 (3550)	32	330	270	200,000	--	200,000	--	200,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4,6-Trichlorophenol	8270 (3550)	20	330	0.2	520	390	11,000	540	390.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dichlorophenol	8270 (3550)	23	330	1	6,100	--	610	--	610.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dimethylphenol	8270 (3550)	35	330	9	41,000	--	41,000	--	41,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dinitrophenol	8270 (3550)	170	1700	0.2	4,100	--	410	--	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,4-Dinitrotoluene (MS)	8270 (3550)	19	330	0.0008	8.4	--	180	--	8.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2,6-Dichlorophenol	8270 (3550)	53	330	Not Available								

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
2,6-Dinitrotoluene	8270 (3550)	19	330	0.0007	8.4	--	180	--	8.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Acetylaminofluorene	8270 (3550)	38	330	Not Available								
2-Chloronaphthalene	8270 (3550)	24	330	Not Available								
2-Chlorophenol (MS)	8270 (3550)	27	330	4	10,000	53,000	10,000	53,000	10,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Methyl phenol (o-Cresol)	8270 (3550)	30	330	15	100,000	--			100,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
2-Methylnaphthalene	8270 (3550)	24	330	7.7	8,200	--	820	--	820.00	IEPA TACO - Class I	No	No
2-Naphthylamine	8270 (3550)	82	330	Not Available								
2-Nitroaniline	8270 (3550)	23	1700	--	--	120	--	7.5	7.50	IEPA TACO - Class I	Yes	No
2-Nitrophenol	8270 (3550)	20	330	Not Available								
2-Picoline	8270 (3550)	82	330	Not Available								
3- and 4-Methyl phenol	8270 (3550)	29	330	Not Available								
3,3'-Dichlorobenzidine	8270 (3550)	420	660	0.007	13	--			13.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
3,3'-Dimethylbenzidine	8270 (3550)	30	1700	Not Available								
3-Methylcholanthrene	8270 (3550)	26	330	Not Available								
3-Nitroanline	8270 (3550)	33	1700	Not Available								
4,6-Dinitro-2-methylphenol	8270 (3550)	200	1700	Not Available								
4-Aminobiphenyl	8270 (3550)	54	330	Not Available								
4-Bromophenyl phenyl ether	8270 (3550)	31	330	Not Available								
4-Chloro-3-methylphenol (MS)	8270 (3550)	34	330	Not Available								
4-Chloroaniline	8270 (3550)	26	660	0.7	8,200	--	820	--	820.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
4-Chlorophenylphenyl ether	8270 (3550)	19	330	Not Available								
4-Nitroaniline	8270 (3550)	17	1700	Not Available								
4-Nitrophenol (MS)	8270 (3550)	210	1700	Not Available								
4-Nitroquinoline-1-oxide	8270 (3550)	166	3300	Not Available								
7,12-Dimethylbenz(a)anthracene	8270 (3550)	31	330	Not Available								
a,a-Dimethylphenethylamine	8270 (3550)	490	6700	Not Available								
Acenaphthene (MS)	8270 (3550)	19	330	570	120,000	--	120,000	--	120,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Acenaphthylene	8270 (3550)	17	330	24	61,000	--			61,000.00	IEPA TACO - Class I	No	No
Acetophenone	8270 (3550)	22	330	Not Available								
Aniline	8270 (3550)	21	330	0.064	1,000	130	8.6	0.064	0.06	IEPA TACO - Class I	No	No
Anthracene	8270 (3550)	23	330	12,000	610,000	--	610,000	--	610,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Aramite	8270 (3550)	60	330	Not Available								
Benzidine	8270 (3550)	83	2700	4.30E-06	0.02	0.02	0.54	0.02	0.02	IEPA TACO - Class I	No	No
Benzo(a)anthracene	8270 (3550)	31	330	2	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Benzo(a)pyrene	8270 (3550)	19	330	8	0.8	--	17	--	0.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzo(b)fluoranthene	8270 (3550)	26	330	5	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzo(g,h,i)perylene	8270 (3550)	23	330	32,000	61,000	--	61,000	--	61,000.00	IEPA TACO - Class I	Yes	No
Benzo(k)fluoranthene	8270 (3550)	36	330	49	78	--	1,700	--	78.00		No	No
Benzoic acid	8270 (3550)	170	1700	400	1,000,000	--	820,000	--	820,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Benzyl alcohol	8270 (3550)	38	330	9.4	610,000	6,900	200,000	6,900	6,900.00	IEPA TACO - Class I	No	No
Bis(2-chloroethoxy) methane	8270 (3550)	24	330	Not Available								
Bis(2-chloroethyl) ether	8270 (3550)	28	330	0.0004	5	0.47	75	1	0.47	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Bis(2-chloroisopropyl) ether	8270 (3550)	35	330	2.4	82,000	1,300			1,300.00	IEPA TACO - Class I	No	No
Bis(2-ethylhexyl) phthalate	8270 (3550)	38	330	3,600	410	31,000	4,100	31,000	410.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Butyl benzyl phthalate	8270 (3550)	27	330	930	410,000	930	410,000	930	930.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Carbazole	8270 (3550)	28	330	0.6	290	--	6,200	--	290.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Chrysene	8270 (3550)	25	330	160	780	--	17,000	--	780.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Diallate	8270 (3550)	56	330	Not Available								
Dibenzo(a,h)anthracene	8270 (3550)	24	330	2	0.8	--	17	--	0.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Dibenzofuran	8270 (3550)	19	330	15	8,200	--	820	--	820.00	IEPA TACO - Class I	No	No
Diethylphthalate	8270 (3550)	22	330	470	1,000,000	2,000	1,000,000	2,000	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dimethoate	8270 (3550)	45	330								No	No
Dimethylphthalate	8270 (3550)	19	330	380	1,000,000	1,300	1,000,000	1,300	1,300.00	IEPA TACO - Class I	Yes	No
Di-n-butylphthalate	8270 (3550)	28	330	2,300	200,000	2,300	200,000	2,300	2,300.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Di-n-octylphthalate	8270 (3550)	31	330	10,000	41,000	10,000	4,100	10,000	4,100.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3550)	83	330	Not Available								
Diphenylamine/ N-nitrosodiphenylamine	8270 (3550)	23	330	20	51,000	--	5,100	--	5,100.00	IEPA TACO - Class I	No	No
Disulfoton	8270 (3550)	55	330	0.097	82	820	8.2	820	8.20	IEPA TACO - Class I	No	No
Ethyl methanesulfonate	8270 (3550)	66	330	Not Available								
Ethyl parathion	8270 (3550)	55	330	Not Available								
Famphur	8270 (3550)	98	330	Not Available								
Fluoranthene	8270 (3550)	26	330	4,300	82,000	--	82,000	--	82,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Fluorene	8270 (3550)	22	330	560	82,000	--	82,000	--	82,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachlorobenzene	8270 (3550)	26	330	2	4	1.8	78	2.6	1.80	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Hexachlorobutadiene	8270 (3550)	20	330	2.9	410	1,000	41	180	41.00	IEPA TACO - Class I	No	No
Hexachlorocyclopentadiene	8270 (3550)	83	330	400	14,000	16	14,000	1.1	1.10	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Hexachloroethane	8270 (3550)	20	330	0.5	2,000	--	2,000	--	2,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Hexachlorophene	8270 (3550)	12500	170000	Not Available								
Hexachloropropene	8270 (3550)	45	330	Not Available								
Indeno(1,2,3-cd)pyrene	8270 (3550)	26	330	14	8	--	170	--	8.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isophorone	8270 (3550)	22	330	8	410,000	4,600	410,000	4,600	4,600.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Isosafrole	8270 (3550)	64	330	Not Available								
m-Dinitrobenzene	8270 (3550)	50	330	Not Available								
Methapyrilene	8270 (3550)	42	67000	Not Available								
Methyl methanesulfonate	8270 (3550)	55	330	Not Available								
Methyl parathion	8270 (3550)	47	330	Not Available								
Naphthalene	8270 (3550)	19	330	12	41,000	270			270.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Nitrobenzene	8270 (3550)	35	330	0.1	1,000	140	1,000	9.4	9.40	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
N-Nitro-o-toluidine	8270 (3550)	32	330	Not Available								
N-Nitrosodiethylamine	8270 (3550)	44	330	Not Available								
N-Nitrosodimethylamine	8270 (3550)	59	330	Not Available								
N-Nitroso-di-N-butylamine	8270 (3550)	52	330	Not Available								
N-Nitrosodiphenylamine/ Diphenylamine	8270 (3550)	23	330	1	1,200	--	25,000	--	1,200.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
N-Nitrosomethylethylamine	8270 (3550)	52	330	Not Available								
N-Nitrosomorpholine	8270 (3550)	66	330	Not Available								
N-Nitrosopiperidine	8270 (3550)	71	330	Not Available								
N-Nitrosopyrrolidine	8270 (3550)	45	330	Not Available								
N-Nitrosos-di-N-propylamine (MS)	8270 (3550)	28	330								No	No
o,o,o-Triethylphosphorothioate	8270 (3550)	72	330	Not Available								
o-Toluidine	8270 (3550)	56	330	Not Available								
p-(Dimethylamino)azobenzene	8270 (3550)	38	330	Not Available								
Pentachlorobenzene	8270 (3550)	64	330	Not Available								
Pentachloronitrobenzene	8270 (3550)	56	330	28	6,100	--	610	--	610.00	IEPA TACO - Class I	No	No
Pentachlorophenol (MS)	8270 (3550)	83	1700	Not Available								
Phenacetin	8270 (3550)	51	330	Not Available								
Phenanthrene	8270 (3550)	29	330	220	61,000	--	61,000	--	61,000.00	IEPA TACO - Class I	No	No

Table 4.2
Data Quality Objective Analysis for Soil

PARAMETER	METHOD	MDL (mg/kg)	RL (mg/kg)	Class I Screening Value (mg/kg)	Ingestion (mg/kg) Industrial-Commercial	Inhalation (mg/kg) Industrial-Commercial	Ingestion (mg/kg) Construction Worker	Inhalation (mg/kg) Construction Worker	Most Conservative (mg/kg)	Ref Description	Soil to GW Screening Value > RL?	Soil Screening Value > RL?
Phenol (MS)	8270 (3550)	30	330	100	1,000,000	--	120,000	--	120,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	No	No
Phorate	8270 (3550)	69	330	0.31	410	1,600	41	710	41.00	IEPA TACO - Class I	No	No
p-Phenylenediamine	8270 (3550)	350	1700	Not Available								
Pronamide	8270 (3550)	63	330	Not Available								
Pyrene (MS)	8270 (3550)	20	330	4,200	61,000	--	61,000	--	61,000.00	Tier 1 Soil Remediation Objectives for Industrial/Commercial Properties 26 Ill. Reg. 2683	Yes	No
Pyridine	8270 (3550)	30	330	0.029	2,000	120,000	2,000	5,200	2,000.00	IEPA TACO - Class I	No	No
Safrole	8270 (3550)	53	330	Not Available								
Sulfotepp	8270 (3550)	55	330	Not Available								
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270 (3550)	45	330	Not Available								
Thionazin	8270 (3550)	42	330	Not Available								
Surrogates												
2-Fluorobiphenyl	8270 (3550)	NA	NA	Not Available								
2-Fluorophenol	8270 (3550)	NA	NA	Not Available								
Nitrobenzene-d5	8270 (3550)	NA	NA	Not Available								
Phenol-d5	8270 (3550)	NA	NA	Not Available								
p-Terphenyl-d14	8270 (3550)	NA	NA	Not Available								
2,4,6- Tribromophenol	8270 (3550)	NA	NA	Not Available								

Notes:

1) Shadded chemicals indicate the chemical is not included in the Superfund Target Compound or Analyte List.

2) N.C. = Not Comparable.

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Metals						
Aluminum	6010 (3010)	1	75-125	<=20	30	200
Antimony	6010 (3010)	1	75-125	<=20	5.1	20
Arsenic	6010 (3010)	1	75-125	<=20	5.2	10
Barium	6010 (3010)	1	75-125	<=20	1.2	10
Beryllium	6010 (3010)	1	75-125	<=20	0.14	4.0
Cadmium	6010 (3010)	1	75-125	<=20	1.0	5.0
Calcium	6010 (3010)	1	75-125	<=20	50	500
Chromium	6010 (3010)	1	75-125	<=20	0.95	10
Cobalt	6010 (3010)	1	75-125	<=20	1.3	10
Copper	6010 (3010)	1	75-125	<=20	1.7	20
Iron	6010 (3010)	1	75-125	<=20	28	50
Lead	6010 (3010)	1	75-125	<=20	3.5	5.0
Magnesium	6010 (3010)	1	75-125	<=20	9.8	500
Manganese	6010 (3010)	1	75-125	<=20	2.0	10
Nickel	6010 (3010)	1	75-125	<=20	1.5	40
Potassium	6010 (3010)	1	75-125	<=20	15	1000
Selenium	6010 (3010)	1	75-125	<=20	5.4	10
Silver	6010 (3010)	1	75-125	<=20	1.0	10
Sodium	6010 (3010)	1	75-125	<=20	300	1000
Thallium	6010 (3010)	1	75-125	<=20	11	25
Tin	6010 (3010)	1	75-125	<=20	6.3	50
Vanadium	6010 (3010)	1	75-125	<=20	1.4	10
Zinc	6010 (3010)	1	75-125	<=20	3.0	20
Mercury (CVAA)	7470	1	80-120	<=20	0.080	0.20
Polychlorinated Biphenyls as Aroclors by GC/EC						
PCB 1016 (MS)	8082 (3520)	1	43-114	<=40	0.14	1.0
PCB 1221	8082 (3520)	1	30-110	<=40	0.50	2.0
PCB 1232	8082 (3520)	1	30-110	<=40	0.22	1.0
PCB 1242	8082 (3520)	1	30-110	<=40	0.22	1.0
PCB 1248	8082 (3520)	1	30-110	<=40	0.33	1.0
PCB 1254	8082 (3520)	1	30-110	<=40	0.20	1.0
PCB 1260 (MS)	8082 (3520)	1	52-121	<=40	0.17	1.0
PCB 1268	8082 (3520)	1	40-130	<=40	0.10	1.0
Surrogates						
Decachlorobiphenyl	8082 (3520)	1	30-150	NA	NA	NA
2,4,5,6-Tetrachloro-m-xylene	8082 (3520)	1	30-150	NA	NA	NA
Chlorinated Pesticides in Groundwater by GC/EC						
4,4'-DDD	8081 (3520)	1	59-141	<=40	0.011	0.10
4,4'-DDE	8081 (3520)	1	44-117	<=40	0.012	0.10
4,4'-DDT (MS)	8081 (3520)	1	39-140	<=40	0.014	0.10
Aldrin (MS)	8081 (3520)	1	37-120	<=40	0.0053	0.050
alpha BHC	8081 (3520)	1	31-112	<=40	0.0086	0.050
alpha Chlordane	8081 (3520)	1	45-120	<=40	0.0068	0.050
beta BHC	8081 (3520)	1	19-131	<=40	0.0057	0.050
Chlorobenzilate	8081 (3520)	1	35-155	<=40	0.19	0.50

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Chlorinated Pesticides in Groundwater by GC/EC						
delta BHC	8081 (3520)	1	39-123	<=40	0.0098	0.050
Dieldrin (MS)	8081 (3520)	1	38-136	<=40	0.010	0.10
Endosulfan I	8081 (3520)	1	40-119	<=40	0.0064	0.050
Endosulfan II	8081 (3520)	1	40-123	<=40	0.011	0.10
Endosulfan sulfate	8081 (3520)	1	54-132	<=40	0.010	0.10
Endrin (MS)	8081 (3520)	1	34-146	<=40	0.010	0.10
Endrin aldehyde	8081 (3520)	1	33-142	<=40	0.017	0.10
Endrin ketone	8081 (3520)	1	37-165	<=40	0.016	0.10
gamma BHC (Lindane) (MS)	8081 (3520)	1	27-115	<=40	0.0055	0.050
gamma Chlordane	8081 (3520)	1	55-123	<=40	0.013	0.050
Heptachlor (MS)	8081 (3520)	1	35-121	<=40	0.0076	0.050
Heptachlor epoxide	8081 (3520)	1	49-120	<=40	0.015	0.050
Isodrin	8081 (3520)	1	24-214	<=40	0.0082	0.050
Kepone	8081 (3520)	1	D-105	<=100	0.12	1.0
Methoxychlor	8081 (3520)	1	53-167	<=40	0.024	0.50
Toxaphene	8081 (3520)	1	39-137	<=40	0.88	5.0
Surrogates						
Non-routine Compounds						
2,4'-DDD	8081 (3520)	1	59-141	<=40	0.073	0.10
2,4'-DDE	8081 (3520)	1	44-117	<=40	0.068	0.10
2,4'-DDT	8081 (3520)	1	39-140	<=40	0.071	0.10
Chlorinated Herbicides by GC/EC						
2,4,5-T (MS)	8151	1	38-138	<=40	0.080	0.50
2,4,5-TP (Silvex) (MS)	8151	1	44-118	<=40	0.033	0.50
2,4-D (MS)	8151	1	45-146	<=40	0.11	0.50
2,4-DB	8151	1	35-140	<=40	0.057	0.50
Dalapon	8151	1	48-187	<=40	0.24	10
Dicamba	8151	1	60-113	<=40	0.048	0.50
Dichlorprop	8151	1	43-106	<=40	0.030	0.50
Dinoseb	8151	1	23-117	<=40	0.40	6.0
MCPA	8151	1	47-131	<=40	12	120
MCPP	8151	1	27-150	<=40	10	120
Pentachlorophenol	8151	1	46-144	<=40	0.18	0.25
Volatiles in Groundwater by GC/MS						
1,1,1,2-Tetrachloroethane	8260 (5030)	1	62-107	<=30	0.53	1.0
1,1,1-Trichloroethane	8260 (5030)	1	70-132	<=30	0.79	1.0
1,1,2,2-Tetrachloroethane	8260 (5030)	1	71-127	<=30	0.21	1.0
1,1,2-Trichloroethane	8260 (5030)	1	75-122	<=30	0.37	1.0
1,1-Dichloroethane	8260 (5030)	1	70-127	<=30	0.56	1.0
1,1-Dichloroethene (MS)	8260 (5030)	1	64-132	<=30	0.93	1.0
1,2,3-Trichloropropane	8260 (5030)	1	60-147	<=30	0.44	1.0
1,2,4-Trichlorobenzene	8260 (5030)	1	48-131	<=30	0.28	1.0
1,2-Dibromo-3-chloropropane	8260 (5030)	1	14-147	<=50	0.65	1.0
1,2-Dibromoethane	8260 (5030)	1	60-118	<=30	0.34	1.0
1,2-Dichlorobenzene	8260 (5030)	1	71-125	<=30	0.21	1.0
1,2-Dichloroethane	8260 (5030)	1	68-130	<=30	0.28	1.0

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
1,2-Dichloroethenes, Total (sum of cis- and trans- isomers)	8260 (5030)	1	70-130	<=30	1.2	2.0
1,2-Dichloropropane	8260 (5030)	1	74-123	<=30	0.26	1.0
1,3-Dichlorobenzene	8260 (5030)	1	70-125	<=30	0.28	1.0
1,4-Dichlorobenzene	8260 (5030)	1	65-127	<=30	0.44	1.0
2-Butanone (methyl ethyl ketone-MEK)	8260 (5030)	1	51-142	<=30	0.72	10
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5030)	1	70-130	<=30	0.40	1.0
2-Hexanone	8260 (5030)	1	58-139	<=30	0.39	10
3-Chloropropene (Allyl chloride)	8260 (5030)	1	D-200	<=100	0.35	1.0
4-Methyl-2-pentanone (MIBK)	8260 (5030)	1	62-130	<=30	0.45	10
Acetone	8260 (5030)	1	20-183	<=50	7.3	25
Acetonitrile	8260 (5030)	1	71-158	<=30	0.23	40
Acrolein	8260 (5030)	1	40-91	<=30	12	20
Acrylonitrile	8260 (5030)	1	46-144	<=30	3.2	20
Benzene (MS)	8260 (5030)	1	74-122	<=30	0.54	1.0
Bromodichloromethane	8260 (5030)	1	74-128	<=30	0.42	1.0
Bromoform	8260 (5030)	1	64-132	<=30	0.36	1.0
Bromomethane	8260 (5030)	1	21-176	<=50	0.93	1.0
Carbon disulfide	8260 (5030)	1	60-130	<=30	0.75	1.0
Carbon tetrachloride	8260 (5030)	1	64-137	<=30	0.91	1.0
Chlorobenzene (MS)	8260 (5030)	1	75-123	<=30	0.41	1.0
Chloroethane	8260 (5030)	1	40-171	<=50	0.89	1.0
Chloroform	8260 (5030)	1	74-124	<=30	0.52	1.0
Chloromethane	8260 (5030)	1	51-133	<=50	0.53	1.0
cis-1,2-Dichloroethene	8260 (5030)	1	69-126	<=30	0.55	1.0
cis-1,3-Dichloropropene	8260 (5030)	1	76-126	<=30	0.25	1.0
Dibromochloromethane	8260 (5030)	1	75-126	<=30	0.40	1.0
Dibromomethane	8260 (5030)	1	70-130	<=30	0.33	1.0
Dichlorodifluoromethane	8260 (5030)	1	70-130	<=50	0.73	1.0
Ethyl methacrylate	8260 (5030)	1	58-101	<=30	0.33	1.0
Ethylbenzene	8260 (5030)	1	77-123	<=30	0.62	1.0
Hexachlorobutadiene	8260 (5030)	1	58-133	<=30	0.50	1.0
Iodomethane	8260 (5030)	1	34-116	<=30	0.96	5.0
Isobutyl alcohol	8260 (5030)	1	39-132	<=30	13	40
m&p-Xylene	8260 (5030)	1	74-123	<=30	1.3	2.0
Methacrylonitrile	8260 (5030)	1	65-110	<=30	9.1	20
Methylene chloride	8260 (5030)	1	67-128	<=30	0.44	5.0
Methylmethacrylate	8260 (5030)	1	57-120	<=30	0.36	1.0
Naphthalene	8260 (5030)	1	58-143	<=30	0.12	5.0
o-Xylene	8260 (5030)	1	76-122	<=30	0.49	1.0
Pentachloroethane	8260 (5030)	1	1-200	<=100	1.0	5.0
Propionitrile (ethylcyanide)	8260 (5030)	1	72-121	<=30	10	20
Styrene	8260 (5030)	1	75-125	<=30	0.42	1.0
Tetrachloroethene	8260 (5030)	1	70-133	<=30	0.75	1.0
Toluene (MS)	8260 (5030)	1	75-122	<=30	0.62	1.0
trans-1,2-Dichloroethene	8260 (5030)	1	67-130	<=30	0.80	1.0
trans-1,3-Dichloropropene	8260 (5030)	1	75-126	<=30	0.36	1.0

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Volatiles in Groundwater by GC/MS						
trans-1,4-Dichloro-2-butene	8260 (5030)	1	26-131	<=50	0.80	2.0
Trichloroethene(MS)	8260 (5030)	1	75-122	<=30	0.71	1.0
Trichlorofluoromethane	8260 (5030)	1	74-165	<=50	0.96	1.0
Vinyl acetate	8260 (5030)	1	47-150	<=30	0.70	2.0
Vinyl chloride	8260 (5030)	1	59-136	<=50	0.92	1.0
Xylenes (total)	8260 (5030)	1	77-121	<=30	1.6	2.0
Semivolatiles in Groundwater by GC/MS						
1,2,4,5-Tetrachlorobenzene	8270 (3520)	1	17-127	<=40	1.2	10
1,2,4-Trichlorobenzene (MS)	8270 (3520)	1	46-99	<=40	0.54	10
1,2-Dichlorobenzene	8270 (3520)	1	42-98	<=40	1.0	10
1,3,5-Trinitrobenzene	8270 (3520)	1	10-123	<=40	0.62	10
1,3-Dichlorobenzene	8270 (3520)	1	38-97	<=40	0.55	10
1,4-Dichlorobenzene(MS)	8270 (3520)	1	40-92	<=40	0.52	10
1,4-Dioxane	8270 (3520)	1	22-134	<=40	2.3	10
1,4-Naphthoquinone	8270 (3520)	1	23-49	<=40	0.67	10
1-Naphthylamine	8270 (3520)	1	10-127	<=40	1.2	10
2,3,4,6-Tetrachlorophenol	8270 (3520)	1	10-151	<=40	0.47	10
2,4,5-Trichlorophenol	8270 (3520)	1	62-119	<=40	0.63	10
2,4,6-Trichlorophenol	8270 (3520)	1	61-118	<=40	0.70	10
2,4-Dichlorophenol	8270 (3520)	1	62-112	<=40	0.66	10
2,4-Dimethylphenol	8270 (3520)	1	51-111	<=40	1.0	10
2,4-Dinitrophenol	8270 (3520)	1	13-176	<=40	5.0	50
2,4-Dinitrotoluene (MS)	8270 (3520)	1	45-140	<=40	0.56	10
2,6-Dichlorophenol	8270 (3520)	1	17-125	<=40	0.69	10
2,6-Dinitrotoluene	8270 (3520)	1	65-124	<=40	0.57	10
2-Chloronaphthalene	8270 (3520)	1	58-111	<=40	0.62	10
2-Chlorophenol (MS)	8270 (3520)	1	54-106	<=40	0.72	10
2-Methyl phenol	8270 (3520)	1	57-110	<=40	0.70	10
2-Methylnaphthalene	8270 (3520)	1	51-110	<=40	0.53	10
2-Naphthylamine	8270 (3520)	1	10-119	<=40	0.64	10
2-Nitroaniline	8270 (3520)	1	60-122	<=40	0.69	50
2-Nitrophenol	8270 (3520)	1	59-114	<=40	0.73	10
2-Picoline	8270 (3520)	1	35-102	<=40	0.87	10
3- and 4-Methyl phenol	8270 (3520)	1	49-114	<=40	0.66	10
3,3'-Dichlorobenzidine	8270 (3520)	1	29-101	<=40	1.0	20
3,3'-Dimethylbenzidine	8270 (3520)	1	10-200	<=40	2.5	20
3-Methylcholanthrene	8270 (3520)	1	10-129	<=40	1.4	10
3-Nitroaniline	8270 (3520)	1	46-144	<=40	0.69	50
4,6-Dinitro-2-methylphenol	8270 (3520)	1	42-155	<=40	1.0	50
4-Aminobiphenyl	8270 (3520)	1	20-105	<=40	0.68	10
4-Bromophenyl phenyl ether	8270 (3520)	1	50-112	<=40	0.85	10
4-Chloro-3-methyl-phenol (MS)	8270 (3520)	1	58-118	<=40	0.55	10
4-Chloroaniline	8270 (3520)	1	22-107	<=100	0.52	20
4-Chlorophenylphenyl ether	8270 (3520)	1	59-112	<=40	0.56	10
4-Nitroaniline	8270 (3520)	1	47-127	<=40	0.85	50
4-Nitrophenol (MS)	8270 (3520)	1	30-139	<=40	3.4	50
4-Nitroquinoline-1-oxide	8270 (3520)	1	30-130	<=40	5.0	20

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Semivolatiles in Groundwater by GC/MS						
5-Nitro-o-toluidine	8270 (3520)	1	10-112	<=40	1.2	10
7,12-Dimethylbenz(a)anthracene	8270 (3520)	1	19-112	<=40	1.3	10
a,a-Dimethylphenethylamine	8270 (3520)	1	10-200	<=40	10	2000
Acenaphthene (MS)	8270 (3520)	1	49-117	<=40	0.64	10
Acenaphthylene	8270 (3520)	1	53-117	<=40	0.58	10
Acetophenone	8270 (3520)	1	54-130	<=40	0.83	10
Aniline	8270 (3520)	1	10-92	<=40	0.65	20
Anthracene	8270 (3520)	1	57-118	<=40	0.88	10
Aramite	8270 (3520)	1	10-168	<=40	1.7	10
Benzo(a)anthracene	8270 (3520)	1	55-119	<=40	0.58	10
Benzo(a)pyrene	8270 (3520)	1	36-128	<=40	1.0	10
Benzo(b)fluoranthene	8270 (3520)	1	44-130	<=40	0.78	10
Benzo(g,h,i)perylene	8270 (3520)	1	45-128	<=40	1.0	10
Benzo(k)fluoranthene	8270 (3520)	1	47-129	<=40	0.73	10
Benzyl alcohol	8270 (3520)	1	54-116	<=40	0.80	10
Bis(2-chloroethoxy) methane	8270 (3520)	1	55-115	<=40	0.78	10
Bis(2-chloroethyl) ether	8270 (3520)	1	48-108	<=40	0.72	10
Bis(2-chloroisopropyl)ether (2,2-Oxybis(1-chloropropane))	8270 (3520)	1	45-114	<=40	0.82	10
Bis(2-ethylhexyl) phthalate	8270 (3520)	1	57-125	<=40	0.98	10
Butyl benzyl phthalate	8270 (3520)	1	62-124	<=40	0.70	10
Carbazole	8270 (3520)	1	60-120	<=40	0.82	10
Chrysene	8270 (3520)	1	56-122	<=40	1.0	10
Diallate	8270 (3520)	1	24-148	<=40	1.6	10
Dibenz(a,h)anthracene	8270 (3520)	1	47-126	<=40	1.0	10
Dibenzofuran	8270 (3520)	1	65-109	<=40	0.69	10
Diethyl phthalate	8270 (3520)	1	61-115	<=40	0.59	10
Dimethoate	8270 (3520)	1	48-77	<=40	0.64	10
Dimethylphthalate	8270 (3520)	1	64-112	<=40	0.59	10
Di-n-butyl phthalate	8270 (3520)	1	58-122	<=40	0.82	10
Di-n-octyl phthalate	8270 (3520)	1	55-128	<=40	0.80	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3520)	1	10-127	<=40	2.5	10
Diphenylamine/ Nitrosodiphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
Disulfoton	8270 (3520)	1	36-82	<=40	0.78	10
Ethyl methanesulfonate	8270 (3520)	1	45-127	<=40	0.90	10
Ethyl parathion	8270 (3520)	1	10-75	<=40	0.89	10
Famphur	8270 (3520)	1	10-248	<=40	2.2	10
Fluoranthene	8270 (3520)	1	51-125	<=40	1.0	10
Fluorene	8270 (3520)	1	43-124	<=40	0.55	10
Hexachlorobenzene	8270 (3520)	1	60-122	<=40	1.1	10
Hexachlorobutadiene	8270 (3520)	1	43-109	<=40	1.0	10
Hexachlorocyclopentadiene	8270 (3520)	1	1-84	<=100	5.0	10
Hexachloroethane	8270 (3520)	1	35-89	<=40	1.0	10
Hexachlorophene	8270 (3520)	1	1-253	<=100	47	5000
Hexachloropropene	8270 (3520)	1	10-117	<=40	1.2	10
Indeno(1,2,3-cd)pyrene	8270 (3520)	1	40-132	<=40	0.62	10
Isophorone	8270 (3520)	1	60-113	<=40	0.67	10

Table 4.3
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	Water Parameters					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (ug/L)**	**RL (ug/L)**
Semivolatiles in Groundwater by GC/MS						
Isosafrole	8270 (3520)	1	26-127	<=40	0.96	10
m-Dinitrobenzene	8270 (3520)	1	10-118	<=40	0.68	10
Methapyrilene	8270 (3520)	1	10-86	<=40	1.9	2000
Methyl parathion	8270 (3520)	1	10-82	<=40	0.78	10
Methylmethanesulfonate	8270 (3520)	1	34-136	<=40	0.85	10
Naphthalene	8270 (3520)	1	42-108	<=40	0.76	10
Nitrobenzene	8270 (3520)	1	57-110	<=40	0.93	10
Nitrosodiphenylamine/ Diphenylamine	8270 (3520)	1	30-130	<=40	0.80	10
N-Nitrosodiethylamine	8270 (3520)	1	50-99	<=40	0.82	10
N-Nitrosodimethylamine	8270 (3520)	1	50-137	<=40	1.1	10
N-Nitrosodi-n-butylamine	8270 (3520)	1	30-123	<=40	0.78	10
N-Nitrosodi-n-propylamine(MS)	8270 (3520)	1	49-135	<=40	0.62	10
N-Nitrosomethylethylamine	8270 (3520)	1	10-279	<=40	2.6	10
N-Nitrosomorpholine	8270 (3520)	1	17-151	<=40	1.1	10
N-Nitrosopiperidine	8270 (3520)	1	21-156	<=40	1.2	10
N-Nitrosopyrrolidine	8270 (3520)	1	52-91	<=40	0.57	10
o,o,o-Triethyl-phosphorothioate	8270 (3520)	1	46-138	<=40	1.3	10
o-Toluidine	8270 (3520)	1	10-129	<=40	0.58	10
p-(Dimethylamino)azobenzene	8270 (3520)	1	10-113	<=40	1.2	10
Pentachlorobenzene	8270 (3520)	1	26-127	<=40	1.0	10
Pentachloronitrobenzene	8270 (3520)	1	10-157	<=40	1.2	10
Pentachlorophenol (MS)	8270 (3520)	1	44-132	<=40	2.5	50
Phenacetin	8270 (3520)	1	18-104	<=40	0.82	10
Phenanthrene	8270 (3520)	1	58-120	<=40	0.74	10
Phenol (MS)	8270 (3520)	1	46-106	<=40	0.99	10
Phorate	8270 (3520)	1	36-113	<=40	0.83	10
p-Phenylenediamine	8270 (3520)	1	10-177	<=40	37	2000
Pronamide	8270 (3520)	1	18-122	<=40	0.72	10
Pyrene(MS)	8270 (3520)	1	49-135	<=40	1.0	10
Pyridine	8270 (3520)	1	10-178	<=40	0.81	50
Safrole	8270 (3520)	1	36-114	<=40	0.99	10
Sulfotepp	8270 (3520)	1	30-117	<=40	0.70	10
Thionazin	8270 (3520)	1	42-93	<=40	0.99	10
Non-Routine Analytes						
Atrazine	8270 (3520)	1	40-132	<=50	0.89	10
Benzaldehyde	8270 (3520)	1	30-150	<=50	1.0	10
1,1-Biphenyl (1,1-Diphenyl)	8270 (3520)	1	53-118	<=50	0.68	10
Caprolactam	8270 (3520)	1	30-150	<=50	0.84	10
3-Nitrophenol	8270 (3520)	1	30-130	<=50	0.78	10

REFERENCES AND NOTES FOR WATER PARAMETERS

A "non-routine analyte" is an analyte that requires advance notice prior to arrival in the laboratory. In general, at least one week notice is required to ensure that the laboratory has sufficient instrument capacity, standards, and reagents to adequately process the samples.

Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics using SW-846 methods, percent recoveries are evaluated only on the subset spike compound lists specified by the methods unless noted in a pre-project plan or QAPP. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable.

ABBREVIATIONS USED IN THE APPENDICES

PARAMETER-refers to the compound, analyte or measurement being tested; the field of testing

METHOD-refers to the reference method used to measure the parameter

REF-a number designation that corresponds to the method references and citations

ACC-accuracy measured as percent recovery

PREC-precision measured as relative percent difference

RL-reporting limit

MDL-method detection limit

D-analyte detected (meets qualitative identification criteria)

ICP-inductively coupled (argon) plasma

ICP-MS-inductively coupled (argon) plasma coupled to a mass spectrometer

GFAA -graphite furnace atomic absorption

CVAA - cold vapor atomic absorption

IC - ion chromatography

GC/HECD - gas chromatograph equipped with a Hall electrolytic conductivity detector

GC/PID - gas chromatograph equipped with a photoionization detector

GC/FID - gas chromatograph equipped with a flame ionization detector

GC/EC - gas chromatograph equipped with an electron capture detector

GC/NPD - gas chromatograph equipped with a nitrogen-phosphorus detector

GC/MS - gas chromatrograph equipped with a mass spectrometer

HPLC - high performance liquid chromatography

SEVERN TRENT STL

REFERENCES AND NOTES FOR APPENDIX A

REF #	REFERENCE
1	Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Update III) USEPA Office of Solid Waste and Emergency Response, Washington, DC.
2	EPA 600/4-79-020:Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Research and Development, Cincinatti, OH, March 1983.
3	EPA 600/R-94/111: "Methods for the Determination of Metals in Environmental Samples" May 1994, Supplement 1.
4	Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association, Washington, DC.
5	EPA 200.9: Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Adsorption Spectrometry; Revision 1.2, April 1991; EPA-EMSL
6	Laboratory SOP ME72:*Silica: Preparation and Analysis*
7	Laboratory SOP GE115:*Ion Chromatography*
8	Florida Department of Environmental Regulation; Chemistry Laboratory Methods Manual, Tallahassee. "Calculation of Un-ionized Ammonia in Fresh Water" Revision 1, 10/03/83.
9	ASTM Method D 4282-89 "Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion." (reapproved 1994)
10	Method 1650: Absorbable Organic Halides by Adsorption in Microcoulometric Titrations; Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Water and Wastewater: U.S. EPA Office of Water, Engineering, and Analysis Division, Washington, D.C.
11	HACH Method 8141, Hydrazine by p-Dimethylaminobenzaldehyde Method. Adapted from ASTM Manual of Industrial Wastes, D1385-78, 376 (1979)
12	EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.
13	Laboratory SOP GE27: Calculation of Total and Organic Nitrogen
14	Method 1664:N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), EPA-821-B-94-004b, April 1995.
15	Method 314.0: Determination of Perchlorate in Drinking Water Using Ion Chromatography, Revision 1.0, November 1999. USEPA Office of Water.
16	American Society for Testing and Materials (ASTM) Method D1498-76 (re-approved in 1981); Laboratory SOP BA72: Oxidation-Reduction Potential (ORP)
17	EPA 600/4-88-039: Methods for the Determination of Organic Compounds in Drinking Water, December, 1988, Revised July, 1991.Methods for the Determination of Organic Compounds in Drinking Water, Supplement I, July 1990. Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, August 1992
18	Code of Federal Regulations, Title 40, Part 136 ; U.S. Government Printing Office: Washington, DC, July 1, 1997.
19	Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater: EPA Method 614, February 1982.
20	Determination of Organophosphorus Pesticides in Wastewater: EPA Method 614.1

SEVERN TRENT STL

REFERENCES AND NOTES FOR APPENDIX A

REF #	REFERENCE
21	Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: EPA Method 622 ; January, 1982.
22	Determination of Thiophosphate Pesticides in Industrial and Municipal Wastewater: EPA Method 622.1; January, 1982.
23	Determination of Chlorinated Herbicides in Industrial and Municipal Wastewater. EPA Method 615, January 1982.
24	Method 680: Determination of Pesticides and PCBs in Water and Soil/Sediment by GC/MS U.S. EPA, Washington, D.C..
25	Method 1653: Chlorinated Phenolics in Water by In-situ Acetylation/GC/MS Determination, Method cp. 86.01, National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Avenue, NY 10016 (July 1986)
26	Method for the Determination of Gasoline Range Organics , State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks
27	FL-PRO Method, "Method for Determination of Petroleum Range Organics," FL DEP, Revision 1, November 1, 1995.
28	Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID ; State of Tennessee Department of Environment and Conservation. Effective May 1, 1999.
29	TNRCC Method for Total Petroleum Hydrocarbons, TNRCC Method 1005 (Revision 03, 6/1/01), State of Texas Method.
30	"NCASI Method DI/HAPS-99.01 Selected HAPS in Condensates by GC/FID. NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
31	"NCASI Method DI/MEOH-94.03 Methanol in Process Liquids by GC/FID" NCASI Southern Regional Center May 2000. National Council for Air and Stream Improvement, Inc., 2000. Methods Manual.
32	RSKSOP 175: Dissolved Oxygen and Methane in Water by GC Headspace Equilibration Technique by Kampbell and Wilson, USEPS, March 1989. Laboratory SOP AR30:*Dissolved Gases in Water*.
33	Method 200.8: Determination of Trace Elements in Water and Wastes by ICP/MS. Revision 5.4 (EMMC Version). USEPA Office of Research and Development (1994)
34	California LUFT (Leaking Underground Fuel Tanks) Method; LUFT Manual: Guidelines for Assessment, Cleanup, and Underground Storage Tank Closure. State of California LUFT Task Force, May 1988.

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL

SOIL PARAMETERS

PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Metals Parameters						
Aluminum	6010 (3050)	1	75-125	<=20	4.5	2.0
Antimony	6010 (3050)	1	75-125	<=20	0.45	2.0
Arsenic	6010 (3050)	1	75-125	<=20	0.67	1.0
Barium	6010 (3050)	1	75-125	<=20	0.30	1.0
Beryllium	6010 (3050)	1	75-125	<=20	0.017	0.40
Boron	6010 (3050)	1	75-125	<=20	1.30	5.0
Cadmium	6010 (3050)	1	75-125	<=20	0.22	0.50
Calcium	6010 (3050)	1	75-125	<=20	2.4	50
Chromium	6010 (3050)	1	75-125	<=20	0.13	1.0
Cobalt	6010 (3050)	1	75-125	<=20	0.17	1.0
Copper	6010 (3050)	1	75-125	<=20	0.17	2.0
Iron	6010 (3050)	1	75-125	<=20	4.2	5.0
Lead	6010 (3050)	1	75-125	<=20	0.21	0.50
Magnesium	6010 (3050)	1	75-125	<=20	1.2	50
Manganese	6010 (3050)	1	75-125	<=20	0.21	1.0
Molybdenum	6010 (3050)	1	75-125	<=20	0.33	1.0
Nickel	6010 (3050)	1	75-125	<=20	0.26	4.0
Potassium	6010 (3050)	1	75-125	<=20	1.3	100
Selenium	6010 (3050)	1	75-125	<=20	0.90	2.5
Silver	6010 (3050)	1	75-125	<=20	0.099	1.0
Sodium	6010 (3050)	1	75-125	<=20	50	100
Strontium	6010 (3050)	1	75-125	<=20	0.15	1.0
Thallium	6010 (3050)	1	75-125	<=20	1.30	2.5
Tin	6010 (3050)	1	75-125	<=20	4.0	10
Titanium	6010 (3050)	1	70-130	<=20	0.040	1.0
Vanadium	6010 (3050)	1	75-125	<=20	0.14	1.0
Zinc	6010 (3050)	1	75-125	<=20	0.75	2.0
Mercury	7471	1	80-120	<=20	0.0040	0.020
General Chemistry Parameters						
Ammonia (as N)	350.1(EPA-CE:3-140)	3/4	75-125	<=30	0.075	0.15
Polychlorinated Biphenyls as Aroclors by GC/EC (Sonication Extraction)						
PCB-1016	8082 (3550)	1	34-128	<=50	6.7	33
PCB 1221	8082 (3550)	1	30-130	<=50	6.8	67
PCB 1232	8082 (3550)	1	30-130	<=50	6.2	33
PCB-1242	8082 (3550)	1	30-130	<=50	7.5	33
PCB-1248	8082 (3550)	1	30-130	<=50	8.0	33
PCB-1254	8082 (3550)	1	30-130	<=50	5.2	33
PCB-1260	8082 (3550)	1	28-168	<=51	6.4	33
PCB-1268	8082 (3550)	1	30-150	<=52	11	33
Surrogates						
2,4,5,6-Tetrachloro-m-xylene	8082 (3550)	1	30-150	NA	NA	NA
Decachlorobiphenyl	8082 (3550)	1	30-150	NA	NA	NA

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (mg/kg)**	**RL (mg/kg)**
Chlorinated Pesticides by GC/EC (Sonication Extraction)						
4,4'-DDD	8081 (3550)	1	35-149	<=50	0.30	3.3
4,4'-DDE	8081 (3550)	1	35-122	<=50	0.30	3.3
4,4'-DDT (MS)	8081 (3550)	1	24-171	<=50	0.27	3.3
Aldrin (MS)	8081 (3550)	1	26-139	<=50	0.14	1.7
alpha Chlordane	8081 (3550)	1	42-125	<=50	0.17	1.7
alpha-BHC	8081 (3550)	1	23-127	<=50	0.52	1.7
beta-BHC	8081 (3550)	1	22-134	<=50	0.47	1.7
Chlorobenzilate	8081 (3550)	1	73-201	<=50	3.8	17
delta-BHC	8081 (3550)	1	43-123	<=50	0.23	1.7
Dieldrin (MS)	8081 (3550)	1	29-146	<=50	0.35	3.3
Endosulfan I	8081 (3550)	1	31-124	<=50	0.16	1.7
Endosulfan II	8081 (3550)	1	31-127	<=50	0.27	3.3
Endosulfan sulfate	8081 (3550)	1	55-136	<=50	0.37	3.3
Endrin (MS)	8081 (3550)	1	45-148	<=50	0.32	3.3
Endrin aldehyde	8081 (3550)	1	36-123	<=50	0.65	3.3
Endrin ketone	8081 (3550)	1	47-156	<=50	0.32	3.3
gamma Chlordane	8081 (3550)	1	37-152	<=50	0.22	1.7
gamma-BHC (Lindane) (MS)	8081 (3550)	1	16-144	<=50	0.14	1.7
Heptachlor (MS)	8081 (3550)	1	19-150	<=50	0.32	1.7
Heptachlor epoxide	8081 (3550)	1	43-132	<=50	0.21	1.7
Isodrin	8081 (3550)	1	14-188	<=50	0.33	3.3
Kepone	8081 (3550)	1	10-65	<=50	6.7	170
Methoxychlor	8081 (3550)	1	13-208	<=50	0.47	17
Technical Chlordane	8081 (3550)	1	41-177	<=50	3	17
Toxaphene	8081 (3550)	1	36-159	<=50	12	170
Surrogates						
Tetrachloro-m-xylene	8081 (3550)	1	30-150	NA	NA	NA
Decachlorobiphenyl	8081 (3550)	1	30-150	NA	NA	NA
Non-routine Compounds						
2,4'-DDD	8081 (3550)	1	35-149	<=50	3.3	3.3
2,4'-DDE	8081 (3550)	1	35-122	<=50	3.3	3.3
2,4'-DDT	8081 (3550)	1	24-171	<=50	3.3	3.3
Chlorinated Herbicides by GC/EC						
2,4-D (MS)	8151	1	53-135	<=50	0.97	8.3
2,4-DB	8151	1	31-106	<=50	1.0	8.3
2,4,5-T (MS)	8151	1	43-121	<=50	0.56	8.3
2,4,5-TP (Silvex) (MS)	8151	1	38-119	<=50	0.52	8.3
Dalapon	8151	1	59-164	<=50	10	330
Dicamba	8151	1	59-107	<=50	1.5	8.3
Dichlorprop	8151	1	48-96	<=50	1.8	8.3
Dinoseb	8151	1	31-92	<=50	14	100
MCPA	8151	1	57-117	<=50	42	2000
MCPP	8151	1	30-140	<=50	220	2000
Pentachlorophenol	8151	1	71-109	<=50	0.98	8.3
Surrogates						
2,4-Dichlorophenyl acetic acid	8151	1	34-127	NA	NA	NA
Non-Routine Compounds						
Picloram	8151	1	30-150	<=50	2.6	8.3

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (mg/kg)**	**RL (mg/kg)**
Volatiles by GC/MS						
1,1,1,2-Tetrachloroethane	8260 (5035)	1	70-130	<=50	0.45	5.0
1,1,1-Trichloroethane	8260 (5035)	1	58-139	<=50	0.50	5.0
1,1,2,2-Tetrachloroethane	8260 (5035)	1	64-130	<=50	0.60	5.0
1,1,2-Trichloro-1,2,2-trifluoroethane	8260 (5035)	1	70-130	<=50	1.2	5.0
1,1,2-Trichloroethane	8260 (5035)	1	76-120	<=50	0.54	5.0
1,1-Dichloroethane	8260 (5035)	1	43-157	<=50	0.4	5.0
1,1-Dichloroethene (MS)	8260 (5035)	1	52-143	<=50	0.56	5.0
1,1-Dichloropropene	8260 (5035)	1	76-126	<=50	0.64	5.0
1,2,3-Trichlorobenzene	8260 (5035)	1	29-169	<=50	0.38	5.0
1,2,3-Trichloropropane	8260 (5035)	1	33-210	<=50	0.87	5.0
1,2,4-Trichlorobenzene	8260 (5035)	1	49-152	<=50	0.46	5.0
1,2,4-Trimethylbenzene	8260 (5035)	1	74-133	<=50	0.4	5.0
1,2-Dibromo-3-chloropropane (DBCP)	8260 (5035)	1	21-180	<=100	1.0	10
1,2-Dibromoethane (EDB)	8260 (5035)	1	76-130	<=50	0.73	5.0
1,2-Dichlorobenzene	8260 (5035)	1	81-122	<=50	0.53	5.0
1,2-Dichloroethane	8260 (5035)	1	65-133	<=50	0.59	5.0
1,2-Dichloroethenes (total)	8260 (5035)	1	35-154	<=50	0.62	10
1,2-Dichloropropane	8260 (5035)	1	77-118	<=50	0.42	5.0
1,3,5-Trimethylbenzene	8260 (5035)	1	72-124	<=50	0.34	5.0
1,3-Dichlorobenzene	8260 (5035)	1	84-120	<=50	0.5	5.0
1,3-Dichloropropane	8260 (5035)	1	73-146	<=50	0.56	5.0
1,4-Dichlorobenzene	8260 (5035)	1	67-133	<=50	0.47	5.0
2,2-Dichloropropane	8260 (5035)	1	28-187	<=50	0.59	5.0
2-Butanone (Methyl Ethyl Ketone-MEK)	8260 (5035)	1	30-149	<=50	1.8	25
2-Chloro-1,3-butadiene (Chloroprene)	8260 (5035)	1	70-130	<=50	2.1	5.0
2-Chloroethyl vinyl ether	8260 (5035)	1	D-200	<=100	50.0	50
2-Chlorotoluene	8260 (5035)	1	49-219	<=50	0.5	5.0
2-Hexanone	8260 (5035)	1	30-148	<=50	2.0	25
3-Chloropropene (Allyl chloride)	8260 (5035)	1	40-165	<=50	2.3	5.0
4-Chlorotoluene	8260 (5035)	1	45-218	<=50	0.57	5.0
4-Methyl-2-pentanone (MIBK)	8260 (5035)	1	29-150	<=50	1.9	25
Acetone	8260 (5035)	1	28-143	<=100	2.6	50
Acetonitrile	8260 (5035)	1	61-148	<=50	53	200
Acrolein	8260 (5035)	1	1-123	<=100	23	100
Acrylonitrile	8260 (5035)	1	44-125	<=50	16	100
Benzene (MS)	8260 (5035)	1	79-118	<=50	0.52	5.0
Bromobenzene	8260 (5035)	1	77-147	<=50	0.4	5.0
Bromochloromethane	8260 (5035)	1	63-136	<=50	0.47	5.0
Bromodichloromethane	8260 (5035)	1	74-128	<=50	0.42	5.0
Bromoform	8260 (5035)	1	62-137	<=50	0.62	5.0
Bromomethane	8260 (5035)	1	26-160	<=100	4.3	5.0
Carbon disulfide	8260 (5035)	1	32-157	<=50	0.4	5.0
Carbon tetrachloride	8260 (5035)	1	62-140	<=50	0.36	5.0
Chlorobenzene (MS)	8260 (5035)	1	81-120	<=50	0.84	5.0
Chloroethane	8260 (5035)	1	20-140	<=100	1.0	5.0
Chloroform	8260 (5035)	1	77-125	<=50	0.36	5.0
Chloromethane	8260 (5035)	1	42-140	<=100	0.42	5.0
cis-1,2-Dichloroethene	8260 (5035)	1	69-131	<=50	0.30	5.0
cis-1,3-Dichloropropene	8260 (5035)	1	71-123	<=50	0.45	5.0
Dibromochloromethane	8260 (5035)	1	67-135	<=50	0.5	5.0
Dibromomethane	8260 (5035)	1	71-134	<=50	0.42	5.0

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL

SOIL PARAMETERS

PARAMETER	METHOD	REF	ACC (%REC)	PREC (%RPD)	MDL (mg/kg)	RL (mg/kg)
Volatiles by GC/MS						
Dichlorodifluoromethane	8260 (5035)	1	70-130	<=100	0.90	5.0
Ethyl methacrylate	8260 (5035)	1	66-152	<=50	1.2	5.0
Ethylbenzene	8260 (5035)	1	82-118	<=50	0.6	5.0
Hexachlorobutadiene	8260 (5035)	1	29-135	<=100	0.68	5.0
Iodomethane	8260 (5035)	1	35-162	<=50	1.3	5.0
Isobutyl alcohol	8260 (5035)	1	74-136	<=50	83	200
Isopropylbenzene	8260 (5035)	1	75-134	<=50	0.35	5.0
m&p-Xylene	8260 (5035)	1	74-121	<=50	1.5	5.0
Methacrylonitrile	8260 (5035)	1	60-142	<=50	31	100
Methyl methacrylate	8260 (5035)	1	54-155	<=50	2.0	5.0
Methyl t-butyl ether (MTBE)	8260 (5035)	1	37-168	<=50	0.69	50
Methylene chloride	8260 (5035)	1	54-150	<=100	0.87	5.0
Naphthalene	8260 (5035)	1	42-250	<=50	1.8	5.0
n-Butylbenzene	8260 (5035)	1	59-120	<=50	0.42	5.0
n-Propylbenzene	8260 (5035)	1	67-134	<=50	0.47	5.0
o-Xylene	8260 (5035)	1	74-122	<=50	0.44	5.0
Pentachloroethane	8260 (5035)	1	1-195	<=100	1.4	25
p-Isopropyltoluene	8260 (5035)	1	39-141	<=50	0.39	5.0
Propionitrile (ethylcyanide)	8260 (5035)	1	58-142	<=50	30	100
sec-Butylbenzene	8260 (5035)	1	60-128	<=50	0.45	5.0
Styrene	8260 (5035)	1	80-118	<=50	0.53	5.0
tert-Butylbenzene	8260 (5035)	1	62-140	<=50	0.52	5.0
Tetrachloroethene	8260 (5035)	1	79-132	<=50	0.91	5.0
Toluene (MS)	8260 (5035)	1	80-118	<=50	0.69	5.0
trans-1,2-Dichloroethene	8260 (5035)	1	35-154	<=50	0.37	5.0
trans-1,3-Dichloropropene	8260 (5035)	1	75-126	<=50	0.45	5.0
trans-1,4-Dichloro-2-butene	8260 (5035)	1	27-150	<=100	3.6	10
Trichloroethene (MS)	8260 (5035)	1	80-122	<=50	4.4	5.0
Trichlorofluoromethane	8260 (5035)	1	38-146	<=100	0.4	5.0
Vinyl acetate	8260 (5035)	1	1-184	<=100	0.83	10
Vinyl chloride	8260 (5035)	1	34-154	<=100	0.59	5.0
Xylenes (total)	8260 (5035)	1	74-122	<=50	1.9	10
Surrogates						
p-Bromofluorobenzene	8260 (5035)	1	68-121	NA	NA	NA
Dibromofluoromethane	8260 (5035)	1	66-127	NA	NA	NA
Toluene-d8	8260 (5035)	1	65-128	NA	NA	NA
Non-Routine Compounds						
1-Chlorohexane	8260 (5035)	1	70-130	<=50	0.72	5.0
Cyclohexane	8260 (5035)	1	70-130	<=50	10	10
Cyclohexanone	8260 (5035)	1	70-130	<=50	15	50
Diethyl ether	8260 (5035)	1	70-130	<=50	0.78	10
Furan	8260 (5035)	1	70-130	<=50	0.36	5.0
Methyl acetate	8260 (5035)	1	70-130	<=50	1.6	10
Methyl cyclohexane	8260 (5035)	1	70-130	<=50	0.48	10
Tetrahydrofuran	8260 (5035)	1	70-130	<=50	0.36	10
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
1,2,4,5-Tetrachlorobenzene	8270 (3550)	1	37-124	<=50	31	330
1,2,4-Trichlorobenzene (MS)	8270 (3550)	1	36-98	<=50	21	330
1,2-Dichlorobenzene	8270 (3550)	1	35-93	<=50	23	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (mg/kg)**	**RL (mg/kg)**
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
1,2-Diphenyl hydrazine	8270 (3550)	1	22-129	<=50	22	330
1,3,5-Trinitrobenzene	8270 (3550)	1	1-131	<=100	82	330
1,3-Dichlorobenzene	8270 (3550)	1	34-90	<=50	26	330
1,4-Dichlorobenzene (MS)	8270 (3550)	1	32-90	<=50	22	330
1,4-Dioxane	8270 (3550)	1	1-156	<=100	66	330
1,4-naphthoquinone	8270 (3550)	1	1-122	<=100	39	330
1-Methylnaphthalene	8270 (3550)	1	12-128	<=50	29	330
1-Naphthylamine	8270 (3550)	1	1-47	<=100	82	330
2,4,5-Trichlorophenol	8270 (3550)	1	46-116	<=50	32	330
2,4,6-Trichlorophenol	8270 (3550)	1	44-113	<=50	20	330
2,4-Dichlorophenol	8270 (3550)	1	43-108	<=50	23	330
2,4-Dimethylphenol	8270 (3550)	1	40-112	<=50	35	330
2,4-Dinitrophenol	8270 (3550)	1	1-131	<=50	170	1700
2,4-Dinitrotoluene (MS)	8270 (3550)	1	32-128	<=50	19	330
2,6-Dichlorophenol	8270 (3550)	1	20-138	<=50	53	330
2,6-Dinitrotoluene	8270 (3550)	1	38-128	<=50	19	330
2-Acetylaminofluorene	8270 (3550)	1	1-126	<=100	38	330
2-Chloronaphthalene	8270 (3550)	1	41-110	<=50	24	330
2-Chlorophenol (MS)	8270 (3550)	1	36-99	<=50	27	330
2-Methyl phenol (o-Cresol)	8270 (3550)	1	38-107	<=50	30	330
2-Methylnaphthalene	8270 (3550)	1	39-104	<=50	24	330
2-Naphthylamine	8270 (3550)	1	1-51	<=100	82	330
2-Nitroaniline	8270 (3550)	1	38-124	<=50	23	1700
2-Nitrophenol	8270 (3550)	1	38-104	<=50	20	330
2-Picoline	8270 (3550)	1	19-76	<=50	82	330
3- and 4-Methyl phenol	8270 (3550)	1	37-106	<=50	29	330
3,3'-Dichlorobenzidine	8270 (3550)	1	1-118	<=50	420	660
3,3'-Dimethylbenzidine	8270 (3550)	1	1-58	<=100	30	1700
3-Methylcholanthrene	8270 (3550)	1	1-151	<=100	26	330
3-Nitroanline	8270 (3550)	1	19-118	<=50	33	1700
4,6-Dinitro-2-methylphenol	8270 (3550)	1	11-142	<=50	200	1700
4-Aminobiphenyl	8270 (3550)	1	10-47	<=50	54	330
4-Bromophenyl phenyl ether	8270 (3550)	1	38-106	<=50	31	330
4-Chloro-3-methylphenol (MS)	8270 (3550)	1	39-113	<=50	34	330
4-Chloroaniline	8270 (3550)	1	7-103	<=50	26	660
4-Chlorophenylphenyl ether	8270 (3550)	1	42-111	<=50	19	330
4-Nitroaniline	8270 (3550)	1	32-130	<=50	17	1700
4-Nitrophenol (MS)	8270 (3550)	1	21-132	<=50	210	1700
4-Nitroquinoline-1-oxide	8270 (3550)	1	1-200	<=100	166	3300
7,12-Dimethylbenz(a)anthracene	8270 (3550)	1	11-128	<=50	31	330
a,a-Dimethylphenethylamine	8270 (3550)	1	1-65	<=100	490	6700
Acenaphthene (MS)	8270 (3550)	1	36-108	<=50	19	330
Acenaphthylene	8270 (3550)	1	41-112	<=50	17	330
Acetophenone	8270 (3550)	1	24-108	<=50	22	330
Aniline	8270 (3550)	1	1-86	<=100	21	330
Anthracene	8270 (3550)	1	46-115	<=50	23	330
Aramite	8270 (3550)	1	1-140	<=100	60	330
Benzidine	8270 (3550)	1	1-95	<=100	83	2700
Benzo(a)anthracene	8270 (3550)	1	46-116	<=50	31	330
Benzo(a)pyrene	8270 (3550)	1	37-120	<=50	19	330
Benzo(b)fluoranthene	8270 (3550)	1	35-122	<=50	26	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (mg/kg)**	**RL (mg/kg)**
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
Benzo(g,h,i)perylene	8270 (3550)	1	41-122	<=50	23	330
Benzo(k)fluoranthene	8270 (3550)	1	36-124	<=50	36	330
Benzoic acid	8270 (3550)	1	10-94	<=100	170	1700
Benzyl alcohol	8270 (3550)	1	30-98	<=50	38	330
Bis(2-chloroethoxy) methane	8270 (3550)	1	38-106	<=50	24	330
Bis(2-chloroethyl) ether	8270 (3550)	1	30-98	<=50	28	330
Bis(2-chloroisopropyl) ether	8270 (3550)	1	16-116	<=50	35	330
Bis(2-ethylhexyl) phthalate	8270 (3550)	1	25-134	<=50	38	330
Butyl benzyl phthalate	8270 (3550)	1	43-127	<=50	27	330
Carbazole	8270 (3550)	1	47-118	<=50	28	330
Chrysene	8270 (3550)	1	46-118	<=50	25	330
Diallate	8270 (3550)	1	24-137	<=50	56	330
Dibenz(a,h)anthracene	8270 (3550)	1	41-124	<=50	24	330
Dibenzofuran	8270 (3550)	1	44-108	<=50	19	330
Diethylphthalate	8270 (3550)	1	41-118	<=50	22	330
Dimethoate	8270 (3550)	1	22-102	<=50	45	330
Dimethylphthalate	8270 (3550)	1	43-114	<=50	19	330
Di-n-butylphthalate	8270 (3550)	1	35-93	<=50	28	330
Di-n-octylphthalate	8270 (3550)	1	43-129	<=50	31	330
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270 (3550)	1	1-114	<=100	83	330
Diphenylamine/ N-nitrosodiphenylamine	8270 (3550)	1	16-113	<=50	23	330
Disulfoton	8270 (3550)	1	31-65	<=50	55	330
Ethyl methanesulfonate	8270 (3550)	1	28-113	<=50	66	330
Ethyl parathion	8270 (3550)	1	28-113	<=50	55	330
Famphur	8270 (3550)	1	1-124	<=100	98	330
Fluoranthene	8270 (3550)	1	41-124	<=50	26	330
Fluorene	8270 (3550)	1	37-113	<=50	22	330
Hexachlorobenzene	8270 (3550)	1	46-115	<=50	26	330
Hexachlorobutadiene	8270 (3550)	1	42-105	<=50	20	330
Hexachlorocyclopentadiene	8270 (3550)	1	20-109	<=50	83	330
Hexachloroethane	8270 (3550)	1	31-88	<=50	20	330
Hexachlorophene	8270 (3550)	1	1-164	<=100	12500	170000
Hexachloropropene	8270 (3550)	1	34-116	<=50	45	330
Indeno(1,2,3-cd)pyrene	8270 (3550)	1	36-133	<=50	26	330
Isophorone	8270 (3550)	1	37-106	<=50	22	330
Isosafrole	8270 (3550)	1	25-133	<=50	64	330
m-Dinitrobenzene	8270 (3550)	1	1-134	<=100	50	330
Methapyrilene	8270 (3550)	1	1-110	<=100	42	67000
Methyl methanesulfonate	8270 (3550)	1	37-108	<=50	55	330
Methyl parathion	8270 (3550)	1	19-60	<=50	47	330
Naphthalene	8270 (3550)	1	34-97	<=50	19	330
Nitrobenzene	8270 (3550)	1	33-106	<=50	35	330
N-Nitro-o-toluidine	8270 (3550)	1	10-100	<=50	32	330
N-Nitrosodiethylamine	8270 (3550)	1	10-111	<=50	44	330
N-Nitrosodimethylamine	8270 (3550)	1	10-132	<=50	59	330
N-Nitroso-di-N-butylamine	8270 (3550)	1	32-99	<=50	52	330
N-Nitrosodiphenylamine/ Diphenylamine	8270 (3550)	1	16-113	<=50	23	330
N-Nitrosomethylethylamine	8270 (3550)	1	22-137	<=50	52	330
N-Nitrosomorpholine	8270 (3550)	1	18-129	<=50	66	330
N-Nitrosopiperidine	8270 (3550)	1	26-125	<=50	71	330
N-Nitrosopyrrolidine	8270 (3550)	1	20-108	<=50	45	330

Table 4.4
Laboratory Control Limits and Detection Limits

SEVERN TRENT STL	SOIL PARAMETERS					
PARAMETER	**METHOD**	**REF**	**ACC (%REC)**	**PREC (%RPD)**	**MDL (mg/kg)**	**RL (mg/kg)**
Semivolatiles (Base-Neutrals/Acids) by GC/MS (Sonication Extraction)						
N-Nitrosos-di-N-propylamine (MS)	8270 (3550)	1	24-108	<=50	28	330
o,o,o-Triethylphosphorothioate	8270 (3550)	1	28-124	<=50	72	330
o-Toluidine	8270 (3550)	1	10-58	<=50	56	330
p-(Dimethylamino)azobenzene	8270 (3550)	1	1-124	<=100	38	330
Pentachlorobenzene	8270 (3550)	1	30-133	<=50	64	330
Pentachloronitrobenzene	8270 (3550)	1	22-127	<=50	56	330
Pentachlorophenol (MS)	8270 (3550)	1	27-116	<=50	83	1700
Phenacetin	8270 (3550)	1	1-117	<=100	51	330
Phenanthrene	8270 (3550)	1	47-114	<=50	29	330
Phenol (MS)	8270 (3550)	1	34-98	<=50	30	330
Phorate	8270 (3550)	1	37-81	<=50	69	330
p-Phenylenediamine	8270 (3550)	1	1-130	<=100	350	1700
Pronamide	8270 (3550)	1	27-84	<=50	63	330
Pyrene (MS)	8270 (3550)	1	36-128	<=50	20	330
Pyridine	8270 (3550)	1	1-107	<=100	30	330
Safrole	8270 (3550)	1	34-119	<=50	53	330
Sulfotepp	8270 (3550)	1	31-102	<=50	55	330
Tetrachlorophenols (2,3,4,5 + 2,3,4,6)	8270 (3550)	1	21-106	<=50	45	330
Thionazin	8270 (3550)	1	21-86	<=50	42	330
Surrogates						
2-Fluorobiphenyl	8270 (3550)	1	38-104	NA	NA	NA
2-Fluorophenol	8270 (3550)	1	36-101	NA	NA	NA
Nitrobenzene-d5	8270 (3550)	1	33-94	NA	NA	NA
Phenol-d5	8270 (3550)	1	38-102	NA	NA	NA
p-Terphenyl-d14	8270 (3550)	1	40-129	NA	NA	NA
2,4,6- Tribromophenol	8270 (3550)	1	27-124	NA	NA	NA
Notes:						
1) Shadded chemicals indicate the chemical is not included in the Superfund Target Compound or Analyte List.						
2) NA = Not Applicable.						

REFERENCES AND NOTES FOR SOIL PARAMETERS

A "non-routine analyte" is an analyte that requires advance notice prior to arrival in the laboratory. In general, at least one week notice is required to ensure that the laboratory has sufficient instrument capacity, standards, and reagents to adequately process the samples.

Accuracy data are presented as recoveries for spikes or surrogates. For routine analysis of organics using SW-846 methods, percent recoveries are evaluated only on the subset spike compound lists specified by the methods unless noted in a pre-project plan or QAPP. An (MS) following the parameter name designates the routine matrix and laboratory control spike compounds. Precision data are presented as relative percent difference (% RPD) and are advisory; i.e., not used for laboratory control. Since reportable levels (above detection limit) for most of the organic parameters may not be detected in all environmental samples, precision is usually based on duplicate spike data and evaluated according to method requirements.

Accuracy and precision control limits are primarily derived from in-house laboratory data. Some accuracy and precision limits have been rounded to the nearest "5". In some cases, method limits may be used in lieu of in-house limits because in-house limits are broader than the method limits or are too broad to be usable.

ABBREVIATIONS USED IN THE APPENDIX

PARAMETER-refers to the compound, analyte, or measurement being tested or performed (the field of test)

METHOD-refers to the reference method used to measure the parameter

REF-a number designation that corresponds to the method references and citations listed below

ACC-accuracy control limits measured as percent recovery

PREC-precision measured as relative percent difference

RL-reporting limit

MDL-method detection limit

D-analyte detected (meets qualitative identification criteria)

ICP-inductively coupled (argon) plasma

ICP-inductively coupled (argon) plasma coupled to a mass spectrometer

GFAA -graphite furnace atomic absorption

CVAA - cold vapor atomic absorption

IC - ion chromatography

GC/HECD - gas chromatograph equipped with a Hall electrolytic conductivity detector

GC/PID- gas chromatograph equipped with a photoionization detector

GC/FID - gas chromatograph equipped with a flame ionization detector

GC/EC - gas chromatograph equipped with an electron capture detector

GC/NPD - gas chromatograph equipped with a nitrogen-phosphorus detector

GC/MS - gas chromatrograph equipped with a mass spectrometer

HPLC -high performance liquid chromatography

SEVERN TRENT STL

REFERENCES AND NOTES FOR SOIL PARAMETERS

REF #	REFERENCE
1	Test Methods for Evaluating Solid Waste, Third Edition, SW-846 (including Update III) USEPA Office of Solid Waste and Emergency Response, Washington, DC.
2	ASTM 3987-85: Standard Test Method for Shake Extraction of Solid Waste with Water. American Society of Testing and Materials(1992).
3	EPA 600/4-79-020:Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Reseach and Development, Cincinnati, OH, March 1983.
4	EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.
5	ASTM D240-87; Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter. American Society of Testing and Materials (1991)
6	Methods of Soil Analysis, American Society of Agronomy, Inc., Number 9, Part 2, page 570, (Walkley-Black Procedure).
7	Determination of Total Organic Carbon in Sediment; USEPA Region II Environmental Services Division Monitoring Management Branch; Edison, NJ.Lloyd Kahn, QA Specialist. 7/10/86
8	Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA Office of Research and Development: Washington, DC August 1993, EPA/600/R-93/100.
9	Method 1664:N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons) EPA-821-B-94-004b, April 1995.
10	Method 314.0: Determination of Perchlorate in Drinking Water Using Ion Chromatography, revision 1.0, November 1999. USEPA Office of Groundwater and Drinking Water.
11	Development of an Analytical Method of the Determination of Acid Volatile Sulfides (AVS) in Sediments; EPA Contract No. 68-03-3534, October 1990.
12	EPA/600/8-78/017: Microbiological Methods for Monitoring the Environment - Water and Wastes, December 1978.
13	Method for the Determination of Gasoline Range Organics , State of Tennessee Department of Environment and Conservation, Division of Underground Storage Tanks
14	FL-PRO Method, "Method for Determination of Petroleum Range Organics," FL DEP, Revision 1, November 1, 1995.
15	Method for the Determination of Extractable Petroleum Hydrocarbons by GC/FID ; State of Tennessee Department of Environment and Conservation. Effective May 1, 1999.
16	TNRCC Method for Total Petroleum Hydrocarbons, TNRCC Method 1005 (Revision 03, 6/1/01), State of Texas Method.
17	Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH
18	Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC,. Method 8280
19	California LUFT (Leaking Underground Fuel Tanks) Method; LUFT Manual: Guidelines for Assessment, Cleanup, and Underground Storage Tank Closure. State of California LUFT Task Force, May 1988.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures used in the redevelopment of wells. The purpose of well development is to remove well drilling fluids, solids, or other particulates that may have been introduced into the formation and/or well or deposited on the boring wall in a recently installed well during drilling and construction activities. This restores the hydraulic conductivity of the aquifer material surrounding the well to near pre-well installation conditions. Properly developed monitoring wells allow for the collection of ground water samples which are chemically and physically representative of the aquifer of concern. The procedure is also applicable to older or improperly developed wells that are suspected of not providing representative groundwater samples. Monitoring wells will be developed by pumping or bailing.

2.0 EQUIPMENT NEEDED

The following items are required to properly develop groundwater monitoring wells:

- Well keys
- Electronic water level indicator
- Calculator
- Field notebook
- Waterproof pen
- Submersible pump (or other appropriate pump), air-lift system or PVC, Teflon or stainless steel bailer (sized appropriately for well)
- Nylon rope or wireline (for deep wells) for bailing
- Surge block (sized appropriately for well)
- PVC or stainless steel pipe for operating surge block (sized appropriately for well)
- Thermometer
- pH meter (with automatic temperature compensation)
- Calibration pH buffer solutions
- Conductivity meter
- Polyethylene or glass container (for field parameter measurements)
- Plastic squeeze bottle filled with deionized water
- 5-gallon bucket
- Drums or other large container for redevelopment water, if required
- Appropriate health and safety equipment as specified in the Health and Safety Plan
- Appropriate decontamination equipment as specified in the decontamination SOP
- Well completion information

- Plastic sheeting, if required
- Generator or 12 volt battery if electric pump is to be used for developing

3.0 PROCEDURES

WELL REDEVELOPMENT PROCEDURE

Well Redevelopment: URS personnel will direct a program for the redevelopment of the well without the use of dispersing agents, acids, or explosives. The objectives of well redevelopment are to: (a) assure that groundwater enters the well screen freely, thus yielding a representative groundwater sample and an accurate water level measurement; (b) remove all water that may have been introduced during drilling and well installation; (c) remove very fine-grained sediment in the filter pack and nearby formation so that groundwater samples are not highly turbid and so that silting of the well does not occur. Redevelopment will consist of surging and bailing or pumping until little or no sediment enters the well. Each monitoring well will be redeveloped until a minimum of five well volumes have been removed and pH, specific conductance, and temperature readings stabilize within 10% over a minimum of two successive readings.

If the addition of water is required to facilitate surging and bailing, only formation water from that well will be used. If this is not practical due to tightness of the formation then only bailing will be performed. In all cases, care will be taken not to collapse well screens during redevelopment activities.

The volume of water required for removal during redevelopment is calculated using the following method:

1. Measure the depth to water in the well from the measuring point. This is usually a notched point on the top of PVC riser pipe which has been surveyed.
2. Measure the total depth of the well from the same measuring point used for measuring the depth to water.
3. Calculate the height of water in the well casing by subtracting the depth of water from the total well depth.
4. Calculate the number of gallons of water corresponding to one well volume. This is done by multiplying the height of water in the well casing by the conversion factor corresponding to the inside diameter of the well casing.

The following equation shall be used to calculate the volume of water to be removed during well evacuation:

a) Volume of water in casing (gal) =
[ ]*water column (ft)*π*(well radius)^2*0.0043

b) Volume of water in annulus (gal) =

[ ]*water column (ft)*π*[(borehole radius)^2-(well radius)^2*0.0043*0.3

Multiply the volume of one well casing volume, plus the annulus volume if required, by [ ] to obtain the minimum volume of water to be evacuated.

During the well redevelopment activities, field measurements of temperature, pH, and specific conductivity are made, and the clarity, color, any presence of odors, and other comments regarding water quality are noted in the field notebook and on the well development log. The date, time, and volume of water removed is also recorded at this time. Measurements of pH, conductivity and temperature along with observations will be recorded as each well volume of water is removed. A sample of water will be collected for measurement of pH, conductivity, and temperature at the beginning of well redevelopment in order to establish a baseline for comparison with the water quality as well redevelopment proceeds.

3.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on well redevelopment and also provide a permanent record. These observations and data will be recorded with waterproof ink in a bound weatherproof field book with consecutively numbered pages and on the well redevelopment form attached.

As part of the redevelopment process, the following information will be recorded in the field book:

- Date
- Time
- Weather
- Personnel
- Well designation
- Well location
- Date(s) and time of well redevelopment
- Static water level from top of well casing before and after redevelopment
- Volume of water in well prior to redevelopment
- Volume of water removed and time of removal
- Depth from top of well casing to bottom of well
- Screen length
- Depth from top of well casing to top of sediment inside well, if present, before and after redevelopment

- Field measurements of pH, conductivity, and temperature taken during and after redevelopment
- Physical character of removed water throughout redevelopment (color, odor, and turbidity)
- Type and size/capacity of pump and/or bailer
- Description of redevelopment technique
- Decontamination observations
- Instrument calibration record

1.0 OBJECTIVE

This document defines the standard procedure for measuring water levels in wells. This Standard Operating Procedure (SOP) serves as a supplement to the sampling plan. This procedure describes equipment and field procedures necessary to collect water level measurements. The well locations and frequency of measurement are specified in the plan. This procedure is intended to be used together with the plan and other SOPs.

2.0 EQUIPMENT NEEDED

The equipment necessary to measure water levels includes:

- Solinst Model 101 water level meter or equivalent
- Two 5-gal buckets (with lids) or equivalent for decontamination
- Decontamination brushes
- Alconox soap
- Deionized or distilled water
- Potable water
- Spray bottle
- Field data sheets
- Field notebook
- Appropriate health and safety equipment

3.0 PROCEDURE

Appropriate health and safety equipment, as described in the Health and Safety Plan (HSP) should be worn during well opening, well measurement, and decontamination.

- The water level probe shall be decontaminated prior to use in each monitoring well.
- Observations concerning the well pad, surface or protective casing and other well conditions will be documented in the field notebook.
- The depth of the static water level and the total depth of the well will be measured using an electric water level meter. The measuring point for all the wells shall be the top of stainless steel monitoring well casing. If a reference mark is not found, then all well readings will be referenced to the north rim of the monitoring well riser pipe for standardization.
- The static water level and the total depth of the well shall be measured, recorded

on the water level data sheet, and then immediately rechecked.

- All columns of field data sheets shall be completed, including time of measurement. If measurements are taken over a several-day period, the date of each measurement should be clearly indicated on the form.
- Care shall be taken to verify the readings during each water level measurement period. Any significant changes in water level will be noted by comparing the most recent measurement with past measurements.
- After any measurement is taken, the water level probe shall be decontaminated.
- The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements.

3.2 DOCUMENTATION

Field data sheets or field notebooks will include date, time, well number, total well depth, water level, static water elevation, and comments. The data sheets or notebook shall be neat and legible, and shall be signed and dated by the person completing the page.

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) and necessary equipment for collection of groundwater samples in monitoring wells, extraction wells, or piezometers using low-flow techniques. The term "low flow" refers to the velocity that the groundwater is removed from the soil formation immediately adjacent to the well screen.

In this technique, in order to withdraw water from within the well screen and to lessen drawdown, a pump that minimizes disturbance to the groundwater is operated at a low flow rate. The well is only purged within the screened interval until specific parameters have stabilized and as according to the site-specific work plan. Therefore, the groundwater samples collected are representative of the water bearing formation and hydraulically isolated from the water in the casing. The need to purge three well volumes, as required in traditional techniques, is not necessary with low flow purging and sampling. The low flow procedure described in this SOP is not necessarily applicable for every site or for wells screened in materials with very low permeability.

2.0 EQUIPMENT NEEDED

Equipment used during well purging and sampling:

- Well installation forms and boring logs for well being sampled
- Well keys
- Disposable latex or nitrile gloves
- Assorted tools (knife, screwdriver, etc.)
- New synthetic rope
- Pump and required accessories (described in more detail in following section)
- Electronic water level indicator with 0.01-foot increments
- Graduated cylinder
- Temperature meter
- pH meter (with automatic temperature compensation)
- Conductivity meter
- Turbidity meter
- Dissolved oxygen (DO) meter
- Oxidation reduction potential (ORP) meter
- Flow-through cell
- Calibration fluids

- Paper towels or Kimwipes
- Calculator
- Bound field logbook (logbook)
- Waterproof pen and permanent marker
- Plastic buckets
- 55-gallon drums or truck-mounted tank
- Plastic sheeting
- Appropriate decontamination equipment
- Cooler with ice
- Sample containers and labels
- Groundwater sampling form
- Chain-of-Custody form
- Appropriate health and safety equipment (e.g., photoionization detector (PID))

3.0 PROCEDURE

This section provides the step-by-step procedure for collecting groundwater samples in the field. Observations made during groundwater purging and sampling should be recorded in a logbook in accordance with the work plan.

A. Any equipment used in the sampling procedure that could contact groundwater should be properly decontaminated before each use.

B. Equipment should be calibrated based on the manufacturers' instructions. The frequency of calibration should be specified in the site-specific work plan. According to "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (Unites States Environmental Protection Agency (USEPA), 1996), pH calibration should be performed with at least two buffers that bracket the expected range of values. Dissolved oxygen calibration must be corrected for local barometric pressure readings and elevation.

C. Before well purging begins, the following steps should be performed at each well:

- Inspect the well and surrounding site for security, damage, and evidence of tampering. If damage or tampering is evident, contact the project manager for guidance.
- Place clean plastic sheeting around the well (as necessary)

- Measure ambient volatile organic compounds (VOCs) background levels in the immediate vicinity of the well (i.e., using a PID or a flame ionization detector (FID) per the Health and Safety Plan (HASP).
- Remove the well cap and immediately measure VOCs at the rim of the well and record the readings in the logbook or on the groundwater sampling form. Give the water in the well adequate time to reach equilibrium.

D. After the well has reached equilibrium, the groundwater elevation should be measured to the nearest 1/100-foot. The total well depth and screened interval should be obtained from the well logs. Measuring the total depth prior to sampling should be avoided to prevent resuspension of settled solids in the well casings and to minimize the necessary purge time for turbidity equilibration. The total depth of the well should be confirmed after sampling has been completed.

E. Following measurement of the static groundwater elevation, the appropriate equipment will be slowly and carefully placed in the well. If the wells have light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs) care should be taken to place sampling equipment below or above the NAPL.

Selection of the proper pump is important for low-flow sampling activities. USEPA guidance (1996) notes that dedicated sampling devices capable of purging and sampling are preferred over any other type of device. In addition, the pump must be capable of flow rates between 0.1 and 1.0 liter per minute. A variety of portable sampling devices are available, such as bladder pumps, electrical submersible pumps, gas-driven pumps, inertial lift foot-valve samplers (e.g. check-ball systems), and bailers (a list of pump manufacturers and suppliers is included on pg. 7).

When placing the equipment in the well, the pump intake should be set near the middle or slightly above the middle of the screened interval. If the screen length allows, the pump intake should be at least two feet from the bottom of the screen. Placing the pump intake near the top of the water column can cause stagnant water from the casing to be purged, but placing the pump intake near to the bottom of the well can cause mobilization and entrainment of settled solids from the bottom of the well.

F. Tubing should be connected from the pump to a flow-through cell. Then, calculate the volume of water to fill the flow-through cell. According to American Society for Testing and Materials (ASTM) Standard D 6771 (2002), the frequency of measurements should be equal

to the time required to completely evacuate one volume of the cell. This ensures that independent measurements are made.

G. The pump should be started at a low flow rate, approximately 100 mL/min or the lowest flow rate possible.

H. Water level measurements should continue every two minutes until the measurements indicate that significant drawdown is not occurring. According to ASTM standards (2002), allowable drawdown should never exceed the distance between the top of the well screen and the pump intake. Including a safety factor, also provided by ASTM, drawdown should actually not exceed 25% of this distance. This ensures that water stored in the casing is not purged or sampled. For example, for a 4-foot screen, the pump should be placed at the midpoint of the screen (two feet from the top of the screen to the pump intake). With a safety factor of 25%, this would require drawdown not to exceed six inches.

   Once it has been established that significant drawdown is not occurring, the flow rate may be increased to </= 1 L/min (ASTM, 2002) or, if the flow rate remains the same, water level measurements need only to be taken periodically. However, when the flow rate is increased, water level measurements must continue every two minutes.

   If drawdown surpasses 25% of the distance from the pump intake to the top of the screen even while pumping is occurring at the lowest flow rate possible, samplers should refer to the project specific criteria as found in the appropriate FSP or work plan.

I. Parameters should be documented on the groundwater sampling form and in the logbook. The time between parameter measurements is calculated as follows:

   $T = \frac{V}{Q}$, where

   T = time between measurements (minutes)
   V = volume of the flow-through cell (liters)
   Q = purge flow rate (liters per minute)

J. Sampling should occur as stated in the FSP or work plan. However, in most cases, purging will continue until specific parameters have stabilized over three consecutive flow-through cell volumes. **Table 1** provides guidelines that may be used for parameter stabilization as specified by ASTM Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. These guidelines are to be used in combination with professional judgment.

Table 1. Stabilization Guidelines for Low-Flow Sampling

Parameter	Stabilization Guidelines
	ASTM
DO	+/- 10% or +/-0.2 mg/L, whichever is greatest
ORP	+/- 20 mV
PH	+/- 0.2 units
Conductivity	+/- 3%
Temperature	Not Specified
Turbidity	Not Specified

K. After the relevant parameters have stabilized, the flow-through cell should be disconnected or bypassed for sampling. If, after a considerable number of readings have been taken, parameters have not stabilized, samplers should refer to the work plan or possibly use alternative sampling methods.

L. The flow rate should be adjusted to less than 0.5 L/min for sampling to minimize aeration during the sampling of volatiles.

M. A new pair of disposable latex or nitrile gloves should be put on immediately before sampling.

N. The constituents should be sampled for in the order given below:

- VOCs – Vials should be filled completely so that the water forms a convex meniscus then capped so that no air space exists in the vial. Turn the vial over and tap it to check for bubbles. If air bubbles are observed in the sample vial, remove the lid and attempt to fill the vial two more times, (being careful not to dump out any groundwater currently in the vial). If air bubbles are present twice more, discard the sample vial and repeat the procedure with a new vial. If, after three attempts, air bubbles are still in the vial, make a note of this and place the vial in the cooler.
- Gas sensitive parameters (e.g., ferrous iron, methane, alkalinity)
- Semivolatile organic compounds, pesticides, polychlorinated biphenyls, and herbicides
- Petroleum hydrocarbons
- Metals (unfiltered)
- Explosives
- Any filtered analytes (use in-line filters if possible)

O. Place all samples on ice inside a cooler immediately.

P. Each sample should be identified with the Sample ID, location, analysis number, preservatives, date and time of sampling event, and sampler.

Q. The sample time and constituents to be analyzed for should be recorded in the logbook and on the groundwater sampling form.

R. Chain-of-custody procedures should be started.

S. Sample equipment should be decontaminated.

T. The well sampling order should be dependent on expected levels of contamination in each well, if known, and should be determined prior to sampling. Sampling should progress from the least contaminated to the most contaminated well. Quality assurance/quality control (QA/QC) samples should be collected during groundwater sampling as required in the work plan and/or QAPP.

LIST OF SUPPLIERS WHO PROVIDE PUMPS SUITABLE FOR LOW-FLOW SAMPLING:

Field Environmental. 1-800-3930-4009. www.fieldenvironmental.com. Pumps: peristaltic, QED bladder pumps, Fultz rotor pump, control boxes, compressors, etc.

QED. 1-800-624-2026. www.micropurge.com. Pumps: bladder pumps, flow cell, compressors, etc.

Fultz Pumps. 1-717-248-2300. www.fultzpumps.com.

REFERENCES

ASTM 2002, Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations, ASTM D6771-02, American Society for Testing and Materials. West Conshohocken, PA.

Nielsen, David and Nielsen, Gillian. Technical Guidance on Low-Flow Purging and Sampling and Minimum-Purge Sampling. Second Edition. NEFS-TG001-02. April 2002.

USEPA. 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. EPA/540/S-95/504. OSWER, April, 1996.

1.0 OBJECTIVE

This document defines the standard protocols for sample handling, documentation, tracking, and control and custody of environmental samples. This SOP serves as a supplement to the work plan.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for sample control and custody procedures:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as a Chain of Custody (COC) or sample collection sheet
- Field Notebook
- Ice
- Sample Log-in Book
- Clear Tape
- Duct Tape
- Zip-Loc Bags
- Waterproof pens
- Permanent Markers.

3.0 PROCEDURES FOR SAMPLE IDENTIFICATION, HANDLING, AND DOCUMENTATION

Sample Identification

Samples collected during site activities shall have discrete sample identification numbers. These numbers are necessary to identify and track each of the many samples collected for analysis during the life of this project. In addition, the sample identification numbers will be used in the data base to identify and retrieve the analytical results received from the laboratory.

Each sample is identified by a unique code which indicates the site identification number, sample location number, sample matrix identifier, and sample depth.

The sample identification system for groundwater sampling will involve the following

nomenclature "AAA-BB" where:

"AAA" will denote the monitoring well

- GM8- Monitoring Well Location

"BB" will denote QA/QC sample

- EB- equipment blank
- AD- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate
- TB- Trip Blank

The sample identification system for soil will involve the following nomenclature "S-AA-BB-C-D-EEE" where:

"AA" will denote

- ## - Sample Area

"BB" will denote

- ##- Sample Number

"C" will denote

- #- Initial Sample Depth

"D" will denote

- #- Final Sample Depth

"EEE" will denote QA/QC sample

- DUP- analytical duplicate
- MS or MD – Matrix Spike or Matrix Duplicate

For example, S-04-18-1-2 indicates the soil sample was obtained from sample area 4, the North Production Area, for boring number 18, from 1 to 2 ft bgs.

Sample Labeling

Sample labels will be filled out as completely as possible by a designated member of the sampling team prior to beginning field sampling activities each day. The date, time, sampler's

signature, and the last field of the sample identification number should not be completed until the time of sample collection. All sample labels shall be filled out using waterproof ink. At a minimum, each label shall contain the following information:

- Project name and number,
- Sample number identification,
- Initials of sampler,
- Sampling location (if not already encoded in the sample number),
- Required analysis,
- Date and time of sample collection,
- Space for laboratory sample number, and
- Preservative used, if applicable.

Sample Handling and Shipping

The sample containers will be placed in reclosable Ziploc plastic storage bags and wrapped in protective packing material (bubble wrap). Samples will then be placed right side up in a cooler with ice (double bagged using plastic bags). Once the samples are collected, they must remain in the custody of the sampler or another worker from the site. The samples can also remain unattended in a locked vehicle so tampering with the samples will not be possible. Right before shipment, a custody seal should be placed over the opening of the cooler and then the cooler should be taped all the way around with clear packing tape to prevent tampering with the samples. Samples will be hand delivered or shipped by overnight express carrier for delivery to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. A chain-of-custody (COC) form will accompany each cooler. The temperature of all coolers will be measured upon receipt at the laboratory. A temperature blank will be included in each cooler for temperature measurement purposes.

Field Notes

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

URS personnel will keep a bound field notebook while performing sampling and oversight activities on-site. Forms that will be used include: chain-of-custody, boring log, and soil sampling data sheets and field logbook. The field logbooks will record the following:

- Changes to the plan;
- Personnel conducting the site activities, their arrival and departure times and their destination at the site;
- Location where the work was performed;
- Date, time, weather conditions, equipment, and personnel on site;
- Daily information such as:
  - Work accomplished and the current site status,
  - Equipment calibrations, repairs and results, and
  - Site work zones;
- Specific work activities conducted such as
  - Work zone and headspace readings; and
- Incidents and unusual activities that occur on the site such as, but not limited to, accidents, breaches of security, injuries, equipment failures, or weather related problems.

The following sampling-related information will be recorded in the field logbook by the field sampling team:

- Sample number,
- Project identification,
- Sampling location,
- Required analysis,
- Date and time of sample collection,
- Type and matrix of sample,
- Sampling technique,
- Preservative used, if applicable,
- Sampling conditions,
- Observations,
- Initials of the sampler, and
- Samples collected.

Each page in the field books will be signed by the person making the entry at the end of the day, as well as on the bottom of each page. Anyone making entries in another person's field book will sign and date those entries. Any entry which is to be deleted will have a single cross out which is signed and dated. Sufficient information should be recorded to allow the sampling event to be reconstructed without relying on the sampler's memory. Photographic records will be developed through the use of digital photographs, showing pre-sampling and post-sampling conditions at each site.

Procedures to evaluate field data for this project primarily include checking for transcription errors on the part of field crew members and review of field notebooks. This task will be the responsibility of the URS Field Leader, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the notebook.

Sample Chain-of-Custody

During field sampling activities, traceability of the samples must be maintained from the time the samples are collected until the laboratory data is issued. Initial information concerning collection of the samples will be recorded in the field log book as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form.

The sampler will be responsible for initiating and filling out the COC form. The COC will be signed by the sampler or the field person responsible for sample handling when the sampler relinquishes the samples to anyone else. One COC form will be completed for each cooler of samples collected daily and if samples are not hand delivered, the COC will be placed in a Zip-Loc bag and shipped inside the cooler. COC forms will be used to document the transport and receipt of samples from the field to the lab. Information required on a COC includes the following:

- Sampler signature/date/time/affiliation,
- Project number,
- Date and time of collection,
- Sample identification number,
- Sample type and matrix,
- Sample description/location,
- Analyses requested/comments,

- The total number of containers being sent to the lab and number of containers for each sample,
- The appropriate preservative used,
- If any samples are to be placed on hold at the laboratory, this should be clearly indicated on the COC in the comments section,
- Signature of person(s) relinquishing custody, dates, and times,
- Signature of person(s) accepting custody, dates, and times,
- Method of shipment, and
- Shipping air bill number.

The person responsible for delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the samples. Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager. Copies of the COC forms documenting custody changes and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager by the analytical laboratory to be incorporated into the central files.

CHAIN OF CUSTODY RECORD

SHEET___of___

URS CORPORATION

1001 HIGHLAND PLAZA DRIVE WEST, SUITE 300
ST. LOUIS, MISSOURI 63110
314-429-0100

PROJECT NO:	PROJECT NAME:	NO. OF CONTAINERS	CONTAINER DESCRIPTION / ANALYSES REQUESTED	REMARKS
SAMPLER'S: (Signature)				
DATE / TIME	SAMPLE I.D. NUMBER			

RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED BY: (Signature)	DATE / TIME
RELINQUISHED BY: (Signature)	DATE / TIME	RECEIVED AT LAB BY: (Signature)	DATE / TIME
METHOD OF SHIPMENT:		AIRBILL NO:	

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for sample handling, documentation, and tracking. This SOP serves as a supplement to the plan.

2.0 EQUIPMENT NEEDED

The following equipment will be required for this SOP:

- Waterproof coolers (hard plastic or metal)
- Custody Seals
- Field forms such as COC or sample collection sheet
- Field Notebook
- Ice
- Bubble Wrap
- Clear Tape
- Duct Tape
- Zip Loc Bags
- Sample Containers
- Waterproof Pen
- Permanent Marker.

3.0 SAMPLE CONTAINERS

Certified commercially clean sample containers will be obtained from the contract analytical laboratory. The lab will indicate the type of sample to be collected in each bottle type. The work plan lists the appropriate sample containers for the specific analyses require for each project.

4.0 SAMPLE PRESERVATION

Samples will be preserved at the time of the sample collection. Chemical preservatives, if necessary, will be added to the sample containers either by the laboratory prior to shipment to the field, or in the field by sampling personnel.

After sample collection, each container will be labeled and stored on ice at 4°C in an insulated cooler until packed for shipment until packed for shipment to the laboratory. The ice will be double bagged in Zip Loc storage bags. Freezing samples will not be permitted. Any breakable

sample bottles need to be wrapped in protective packing material (bubble wrap) to prevent breakage during shipping.

5.0 SAMPLE HOLD TIMES

Samples will be hand delivered or shipped by overnight express carrier for delivery to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. The hold time varies for each type of analysis. It will be necessary to check with the lab to verify the hold times to determine how frequently samples need to be sent to the lab.

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded using pens with permanent waterproof ink in a permanently bound weatherproof field log book containing consecutively numbered pages.

1.0 OBJECTIVE

This document defines the Standard Operating Procedure (SOP) for decontamination of equipment used in environmental sites.

2.0 EQUIPMENT NEEDED

The following equipment will be needed for decontamination procedures:

- Brushes
- Wash Tubs
- Buckets
- Scrapers, flat bladed
- Hot water – high pressure washer
- Paper towels
- Alconox detergent (or equivalent)
- Potable tap water
- Laboratory-grade deionized or distilled water
- Garden-type water sprayers

2.0 SAMPLING EQUIPMENT DECONTAMINATION PROCEDURES

Sampling equipment will be decontaminated at the sampling location under the following procedures:

- Personnel will wear the proper PPE to reduce the potential for exposure as required by the HASP.
- Partially fill two buckets with potable tap water, and add Alconox detergent to one of the buckets
- Use brushes to wash the sampling equipment (i.e. stainless steel bowls, stainless steel spoons, sampling utility knife, etc)
- Rinse sampling equipment in bucket containing potable tap water
- Rinse clean equipment with water sprayers containing distilled water (or equivalent)
- Place decontaminated equipment in clean area and allow to air dry.

3.0 DRILLING AND HEAVY EQUIPMENT DECONTAMINATION PROCEDURES

Drilling rigs will be decontaminated at a decontamination station located near a staging area. The decontamination station may be a temporary structure, or mobile trailer, capable of collecting all decontamination fluids. The following steps will be used to decontaminate drilling and heavy equipment.

- Personnel will suit up in proper PPE to reduce the potential for exposure as required by the HASP.
- Equipment showing gross impacted soil materials will be scrapped with a flat-bladed scrapper, and material containerized.
- Equipment that cannot be damaged by water, such as a drill rig, augers, drill bits, sampling equipment, shovels, etc, will be washed with a hot water, high-pressure sprayer, the rinsed with potable water.
- Following decontamination, drilling equipment will be placed on the clean drill rig and moved to the next sampling location. If equipment is not immediately used, it should be stored in a clean designated area.

Equipment rinsante samples of the decontaminated sampling equipment may be collected to verify the effectiveness of the decontamination procedures.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) outlines the procedures for soil sample logging. The purpose of soil sample logging is to record all field notes in a detailed and organized fashion.

2.0 PROCEDURES

At the outset of sample logging, the on-site geologist will record field notes with waterproof ink in a bound field notebook. At a minimum, the daily field notes will include:

- Project name and number
- Date and time
- Weather conditions
- Sampler's name
- Project objective(s).

Throughout the sampling round, the following items will be recorded as appropriate:

- Sample location(s)
- Sample identifications
- Limiting field conditions
- Problems encountered.

Unconsolidated soil samples will be described as follows:

- Descriptive information:
  - Color name (Munsell Color Chart) of the logged interval or sample
  - Color notation including chroma, hue, value, and qualifiers

1. Mottling with abbreviations, descriptors, and criteria for descriptions of mottles as identified below

Descriptors for Mottling

Abundance	Size	Contract
f: few (<2%)	fine (<5 mm)	faint
c: common (2%-20%)	medium (5-15 mm)	distinct
m: many (>20%)	coarse (>15 mm)	prominent

2. Degree of saturation (dry, damp, moist, wet, saturated, or combinations); note depth to groundwater table, if observed

3. Degree of density. Count the blows of each 12-inch increment of the sampler (ASTM-1586-84). Use the values and the density table presented below to determine the degree of density.

Degree of Density

Cohesive Clays		Non-cohesive Granular Soils	
<2	very soft	0-4	very loose
2-4	soft	>4-10	loose
>4-8	medium stiff	>10-30	medium dense
>8-15	stiff	>30-50	dense
>15-30	very stiff	>50	very dense
>30	hard		

4. Soil description according to ASTM's Unified Soil Classification System (USC) and by soil structure:

   – ASTM Unified Soil Classification: The Grade Limits and Grade Standards table presented below provides the grade limits and grade names used by engineers according to ASTM standards D422-63 and D643-78.

Grade Limits and Grade Standards

Grade Limits		Grade Names	
mm	inch	US standard sieve series	
			boulders
305	12.0		
			cobbles
76.2	3.0	3.0 inch	
			gravel
4.75	0.19	No. 4	
2.00	0.08	No. 10	
			medium sand
0.425		No. 40	
0.074		No. 200	
			silt
0.005			
			clay size

Source: AGI data sheet 29.2

   – Course-grained soils include clean gravels and sands and silty or clayey gravels and sands with more than 50% retained on the No. 200 sieve. A table of USC symbols and names for coarse-grained soils is presented below.

USCS Symbols and Names for Coarse-grained Soils

USCS Symbol	Typical Names
GW	Well graded gravels, gravel-sand mixtures, little or no fines
GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
GM	Silty gravels, gravel-sand-silt mixtures
GC	Clayey gravels, gravel-sand-clay mixtures
SW	Well graded sands, gravelly sands, little or no fines
SP	Poorly graded sands, gravelly sands, little or no fines
SM	Silty sand, sand-silt mixtures
SC	Clayey sands, sand-clay mixtures

– Fine-grained soils include inorganic and organic silts and clays; gravelly, sandy, or silty clays; and clayey silts with more than 50% passing the No. 200 sieve. A table of USC symbols and names for fine-grained soils is presented here.

USCS Symbols and Names for Fine-grained Soils

USCS Symbol	Typical Names
ML	Inorganic silts and very fine sands, rock flour, silty, or clayey fine sands, or clayey silts with slight plasticity
CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
OL	Organic silts and organic silty clays of low plasticity
MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
CH	Inorganic clays or high plasticity (residual clays), fat clays
OH	Organic clays of medium to high plasticity, organic silts
Pt	Peat and other highly organic soils

– A table of soil descriptors is presented below.

Soil Descriptors

Calcareous:	containing appreciable quantities of calcium carbonate
Fissured:	containing shrinkage cracks, often filled with fine sand or silt, usually more less vertical
Interbedded:	containing alternating layers of different soil types
Intermixed:	containing appreciable, random, and disoriented quantities of varying color, texture, or constituency
Laminated:	containing thin layers of varying color, texture, or constituency
Layer:	thickness greater than 3 inches

Soil Descriptors

Mottled:	containing appreciable random speckles or pockets of varying color, texture, or constituency
Parting:	paper thin
Poorly graded (well sorted):	primarily one grain size, or having a range of sizes with some intermediate size missing
Slickensided:	having inclined planes of weakness that are slick and glossy in appearance and often result in lower unconfined compression cohesion
Split graded:	containing two predominant grain sizes with intermediate sizes missing
Varved:	sanded or layered with silt or very fine sand (cyclic sedimentary couplet)
Well graded (poorly sorted):	containing wide range of grain sizes and substantial amounts of all intermediate particle sizes
Modifiers:	Predominant type - 50% to 100% Modifying type - 12% to 50% With - 5% to 12% Trace - 1% to 5%

5. Degree of plasticity. The following table presents the terms used to denote the various degrees of plasticity of soil that passes the No. 200 sieve.

Degrees of Plasticity

Descriptive Term	Degree of Plasticity	Plasticity Index Range
SILT	none	non-plastic
Clayey SILT	slight	1-5
SILT & CLAY	low	5-10
CLAY & SILT	medium	10-20
Silty CLAY	high	20-40
CLAY	very high	over 40

6. Drilling information:

   – Drill rig manufacturer, model, and driller (if applicable)

   – Geologist or geotechnical engineer

   – Project name, sample point identification, and location

   – Date samples obtained (and times if required)

   – Type of sampler (e.g., split spoon, Shelby, California), measurements or method of advancing boring or equipment, method of driving sampler, and weight of hammer

   – Drill fluids (if applicable)

- Ground surface or grade elevation (if known)
- Depth penetrated and blow counts/6-inch interval of penetration for ASTM 1586-84 and sample number (if applicable)
- Closed hole intervals and advancement (if applicable)
- Recovery
- Strata changes and changes within samples
- Sampling tool behavior
- Drill string behavior
- Use(s) of borehole
- Disposition(s) of residual soil or cuttings
- Signature or sampling of log (as required)

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the procedure to obtain representative subsurface soil samples for geologic logging and physical and chemical laboratory testing.

2.0 EQUIPMENT NEEDED

The following equipment is typically required:

- Hydraulic percussion hammer Geoprobe
- 1 inch diameter by 3 foot length steel probe rods
- Barrel sampler - 2 1/4 in diameter by 4 ft length
- Acetate liners
- Disposable sample retainers
- Photoionization detector (PID, OVM)
- Surveyor's stakes
- Stainless steel pans, knives and plastic Zip-loc bags
- Sample containers
- Decontamination equipment.

3.0 PROCEDURE

The general procedure for using the Geoprobe equipment for sampling is as follows:

1. Locate boring using facility drawings to check utilities
2. Log boring location on site base map
3. Hydraulically push or drive 1 in. diameter probe rods with barrel sampler attached to the first sample depth
4. Remove barrel sampler and retrieve acetate liner. Visually log and classify the soil, select specimen for physical and/or chemical testing. Record information on field data sheets
5. Decontaminate barrel sampler and install new acetate liner
6. Measure VOC concentrations with PID at top of probe hole prior to sampling the next depth interval (if VOCs are a concern)
7. Insert barrel sampler in exiting probe hole and push or drive sampler to the next sample depth, repeat sampling procedure
8. Repeat Geoprobe sampling until the target depth is reached
9. Record total depth
10. Retrieve probe rods

11. Backfill probe hole with bentonite
12. Place survey stake at boring location
13. Record data collected on boring log and log book
14. Decontaminate equipment.

4.0 DECONTAMINATION

Refer to the HSP for personnel decontamination procedures; refer to the equipment decontamination procedures for equipment decontamination procedures.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used for the calibration and use of the Photoionization Detector (PID) for field headspace analysis and health and safety monitoring.

2.0 PURPOSE

The purpose of this procedure is to develop and maintain good quality control in field operations and to create uniformity between field personnel involved with PID use.

3.0 EQUIPMENT NEEDED

PID (probes with 11.7 eV lamp or equivalent) or suitable for site conditions, log book, user's manual, calibration gas.

4.0 PROCEDURE

Calibration:

1. Prior to calibration, check the function switch on the control panel to make sure it is in the "OFF" position. The probe nozzle is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.
2. Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.
3. Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.
4. Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements.
5. Turn the function switch to the "ON" position. In this position, the UV light source should be on. To verify, glance at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to the Instruction Manual.
6. To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

Instrument Daily Calibration:

1. Insert one end of T-tube into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubble meter) attached.
2. Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotameter. The instrument will draw in the volume required for detection with the rotameter indicating excess flow.
3. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder.)
4. Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 6 above.
5. Record all original and readjusted settings in log book.
6. Set the function switch to 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low-range (0-20 ppm) calibration gas cylinder as described above.
7. Do not adjust the span potentiometer. The observed reading should be $\pm$3 ppm of the concentration specified for the low-range calibration gas. If this is not the case, recalibrate the mid-range scale repeating Steps 1 through 6 above. If the low-range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window according to instruction manual. When the observed reading is within the required tolerances, the instrument is fully calibrated.

Instrument Calibration Check:

1. Exit the exclusion zone and turn meter to "ON" position. Check that the meter is reading a value of zero.
2. Insert one end of T-tube into probe and other end into calibration gas. The third end of the T-tube should be attached to a flow meter.
3. Crack the valve on the calibration gas and read the value shown by the instrument. Record the value and calibration gas concentration on a field-data sheet.
4. If the value shown by the instrument is greater than $\pm$20% of the calibration gas concentration, take meter outside of exclusion zone and recalibrate as outlined above.

Sample Measurement:

1. Place function switch in 0-20 ppm range for field monitoring. This will allow for most sensitive, quick response in detecting airborne contaminants.

2. Before entering a contaminated area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated area. Under no circumstance should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.
3. Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measurements on field-data sheet.

   Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected, recalibrate or obtain a different meter and calibrate as outlined above.
4. When finished, reverse Steps 1 through 6 in Instrument Setup section to shut down the instrument.

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) describes the methods to be used in measuring organic vapors emitted from soils collected with a mechanical device or hand augering device. Results will be used as a field screening for volatile organic vapors.

2.0 PURPOSE

The purpose of this procedure is to maintain uniformity between field personnel performing the measurements and to provide representativeness of readings obtained.

3.0 EQUIPMENT NEEDED

Personal protective equipment, PID, wide-mouth sample jars and aluminum foil or polyethylene bags (Ziploc type), rubber bands, field data forms.

4.0 PROCEDURES

1. Samples are collected and placed in wide-mouth sample jars or polyethylene bags (zip-loc type) so that the jars or bags are approximately half full. The jars or bags are labeled to document sample location and depth, time, date, and field personnel collecting the sample.
2. The glass jar is capped with aluminum foil, a rubber band, and the lid, if it will fit or the bag is zipped shut.
3. The air-tight sample container is then allowed to warm for at least 10-15 minutes to allow the liberation of soil gases into the headspace.
4. Calibrate and prepare PID for use.
5. Puncture the aluminum foil or polyethylene bag with the calibrated monitor probe and allow headspace gases to be drawn through the PID unit.
6. Record the highest response obtained on an appropriate sampling log.
7. Remove the punctured foil and seal jar with the proper lid.
8. Allow instrument to return to zero and repeat procedure for next sample.

1.0 OBJECTIVE

Collection of soil samples for VOC analysis that will minimize the loss of contaminants due to volatilization and biodegradation

2.0 EQUIPMENT

The following equipment is required for each sample point.

- One Terra Core™ (or equivalent)
- Two (40-ml VOA vials with sodium bisulfate preservative)
- One (40-ml VOA vial with methanol);
- One 2 or 4-oz glass jar for screening and/or high level analysis, and dry weight conversions (or as specified by laboratory)
- Paper towels
- Indelible pen
- Clear Tape and Labels.

3.0 PROCEDURE

1. The following general procedures are followed for collection of soil samples with the Terra Core™ sampler
2. Remove plunger from package and seat in the handle of the sampler
3. Push the plunger and sampler straight down into a freshly exposed surface of soil until the sample chamber is full (approximately 5 grams of soil)
4. Slowly remove plunger and sampler and inspect bottom of sampler. If sampler is not full, repeat step 3
5. Remove excess soil from the sampler mouth, the soil plug should be flush with the mouth of the sampler
6. Rotate the plunger that was seated in the handle top 90 degrees until it is aligned with the slots in the body
7. Lower the sampler to the top of one of the 40-mil preserved VOA vials and extrude the sample by pushing the plunger down
8. Quickly place the lid back on the 40-ml VOA vial, making sure vial threads are clear of soil and debris
9. Repeat procedures 2 through 8 to fill the other two preserved vials
10. Collect additional soil and place in 2 or 4 oz glass jar to be used for dry weight conversion.
11. Place sample labels on sample containers
12. Secure label with clear tape and place in cooler, keep sample at 4 degrees Celsius

VOLATILE COMPOUNDS BY GC/MS (EPA 8260B)

Approved by:

3 April 2001
Date

Title: Technical Manager, QA

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures that can be used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. The attached quantitation report lists the target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and internal standard assignments.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision criteria for each target compound are listed in Section 5 of the current revisions of the STL Laboratories' *Comprehensive Quality Assurance Plan* and *Corporate Quality Assurance Plan.*

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40C (optional). Five to twenty-five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

2.3 Low-level (nominally <1mg/kg) soil samples are purged at 40C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.

2.4 High level soils (nominally >1mg/kg) and waste samples are extracted with methanol-1mL of methanol per gram of sample. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

2.5 This method is based on the guidance in SW-846 Methods 8260B and 5035.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedure that you do not understand or that will put yourself or others in a potentially hazardous situation.

3.2 Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, eye protection, or other equipment should be used. Standards and highly contaminated samples should be handled in a hood.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst at each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to safely handle these materials.

3.4 The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with STL-SL SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in STL-SL SOP AN70: *Segregation of Low and High Concentration Volatile and Semivolatile Samples.*

4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-terachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.

4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105 C for one hour or replaced.

5.0 SAMPLE COLLECTION, HANDLING, AND PRESERVATION

5.1 Liquid samples are collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4C (less than 6C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2, notify the department supervisor or project manager. If directed by supervisor or project manager, hydrochloric acid may be added through the septum to bring the pH <2. Do not add more than 400uL (0.40mL) of 1:1 HCl to a VOC vial. If pH cannot be adjusted to <=2 without destroying the integrity of the sample, the sample must be analyzed within 7 days of collection.

The holding time for samples preserved with HCl is 14 days for all target compounds. The holding time for un-preserved samples is 7 days.

5.2 Soils: Soils are routinely collected in duplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The hold time of the preserved sample is 14 days from the date of collection. The hold time for frozen samples is 14 days from the date of collection.

5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4C (less than 6C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.

5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4C (less than 6C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

NOTE: Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See STL-SL SOP AN70. If possible, keep the field QC in the same storage refrigerator as the samples.

6.0 APPARATUS AND MATERIALS

The apparatus and materials listed in this section may vary from lab to lab. The items listed are to give guidance and to provide a general overview of the equipment employed in this analysis.

6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator

6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent

6.4 Supelco Vocarb 3000 trap or equivalent, Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments. The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40C, and spinning the stir bar to mix the sample during the purging step.

6.5 Data System compatible with the analytical system

6.6 Microsyringes: 10ul, 25ul, 50ul, 100ul, 250ul, 500ul, 2.5mL

6.7 Gastight syringe: 5mL, 25mL with luerlock tip

6.8 Volumetric flasks: 1.0mL, 10mL, 100mL

6.9 Recommended Columns

J&W DB-624: 60m x 0.32mm ID, 1.8um film
J&W DB-624: 20m x 0.18mm ID, 1.8um film

7.0 REAGENTS

Reagents must be tracked in accordance with STL-SL SOP AN44: *Reagent Traceability.*

7.1 Reagent water-free of volatile contaminants (obtained by purging with inert gas or carbon filtration)

7.2 Methanol-Burdich and Jackson, Purge and Trap grade

7.3 Sodium bisulfate-reagent grade. This salt is hydroscopic and should be stored in a dessicator.

7.4 Soil preservation solution- Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and store the solution in an area free from VOC-especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. All preparation steps must be in accordance with STL-SL SOP AN41: *Standard Material Traceability.*

8.1 Preparation of **Stock Standards** from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from 2000ug/mL to 10000ug/mL. SL-SOP AN43: *Standard Preparation* gives the general instructions for the preparation of the stock solutions from neat materials.

8.2 Preparation of the **Working Standard** from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10-200ug/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The standards and standard concentrations listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document the standard preparation procedures in the standard traceability log.

8.3 Preparation of the **Calibration Standards** from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range of the analytical system.

8.3.1 Add 5.0mL of reagent water to a 5mL-glass syringe or 25ml of reagent water to a 25-ml glass syringe.

8.3.2 Add a known volume of the working standard to 5.0mL or 25ml of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40C. The lab has the option of using blank sand in the calibration standards.

The calibration standards listed in Table 1 are the suggested for routine use. If other "recipes" are used, the lab must document these standard preparation procedures in the standard traceablity log. A 5mL-purge volume may be used for low level (nominal RL of 1ug/L) if the instrument has sufficient sensitivity to detect the targets and the calibration criteria is met.

9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in STL-SL-SOP AN70.

9.1 **Aqueous samples** are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded prior to analysis to determine if the sample has been properly preserved.

9.2 Preparation of Soil Samples (5035)

9.2.1 Remove the Encore samples and the bulk sample from the storage area.

9.2.2 Test an aliquot of the bulk sample for the presence of carbonates.

- Transfer 5g of sample from the bulk sample to a 40mL vial..
- Add 5ml of the sodium bisulfate solution and shake the vial .
- If the sample exhibits effervescence, the Encore samples should be preserved as described above using 5mL of volatile-free water in place of the sodium bisulfate solution and placed in a freezer at –10C. The analytical hold time for frozen samples is 14 days from collection.
- If no effervescence is noted, the Encore samples may be preserved with 5mL soil preservation solution.

9.2.3 Add a stir bar to a vial and weigh the vial and record its tare weight(or tare the vial and stir bar weight by pressing the autotare button).

9.2.4 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (9.2.2), add 5.0mL of reagent water and freeze at –10C.
The hold time is 14 days from collection.

If not, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.

NOTE: A preparation blank is prepared when Encore samples are transferred. The preparation blank contains the same reagents as the samples-either 5mL of reagent water or 5mL of soil preservation solution.

9.3 A methanol extraction is prepared when the concentration of the target compounds (by direct purge) exceeds the working range of the calibration curve. The bulk sample, collected in the 125-mL sample container, can be used to prepare the methanol extraction. Carry out the preparation quickly to minimize the loss of volatiles.

-Mix the sample with a stainless steel spatula and transfer 10g (+/- 0.5g) to a glass vial.

-Add 8uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

$$Ct(ug/kg, dw) = \frac{0.008mL \otimes 2500ug/mL}{0.010g \otimes solids} = 2000ug/kg, dw$$

-Shake the sample for two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

-For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The lab control standard is prepared by adding 8uL of the surrogate spiking solution and 8uL of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze 125uL of the extract.

The matrix spikes are prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) and 8uL of the matrix spiking solution (2500ug/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for two minutes. Analyze 125uL of the extract or a smaller volume if the VOC concentration is high.

-Add 125uL of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with 125uL) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

9.4.1 Mix the sample with a stainless steel spatula and transfer 1g (+/- 0.2g) to a glass vial.

9.4.2 Add 10uL of the surrogate spiking solution (2500ug/mL) to the sample and quickly add 10mL of purge and trap grade methanol. If the sample is completely soluble in the methanol, dilute to a final volume of 10mL. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g , is calculated:

$$Ct(ug/kg) = \frac{0.010mL \otimes 2500ug/mL}{0.0010g \otimes solids} = 25000ug/kg$$

9.4.2 Shake the sample for one minute. Allow the solvent to separate from the solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding 8uL of the surrogate spiking solution (2500ug/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100uL of the extract.

The lab control standard is prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100uL of the extract.

The matrix spikes are prepared by adding 10uL of the surrogate spiking solution (2500ug/mL) and 10uL of the matrix spiking solution (2500ug/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for one minute.

Add 100uL of the extract (or a smaller volume) to 5.0mL of water (or to 25mL if the calibration is based on 25mL). Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

10.0 PROCEDURE

The following instrument conditions are recommended. The actual conditions may vary due to differences in instrumentation. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

10.1 Instrument Conditions

10.1.1 GC Conditions
GC conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. Each lab is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

10.1.1.1 Example GC temperature program

Initial column temperature: 35 C for 3 minutes
Column temperature program 1: 20C per minute
Intermediate column temperature: 70C for 4 minutes
Column temperature program 2: 10C per minute
Final column temperature: 200C for 5.25 minutes

10.1.1.2 Column flow: Approximately 5-10mL/minute helium with a make-up of 20-25mL/minute helium. Total flow into the jet separator should be about 30mL/minute. The vacuum gauge on the jet separator will read about 0.5Torr.

If no jet separator is used and the column is plumbed directly into the source, the column flow should be adjusted to 0.5-1.0ml/min and a split ratio (desorb to column flow) of about 40:1 established. Smaller bore capillary columns (0.18 to 0.32mm) are required if the column is plumbed directly into the source

10.1.1.3 Mass Spectrometer and interface parameters

Jet separator temperature: 240C
Mass spectrometer interface: 240C
Mass spectrometer source temperature: factory set at 300C
range: 35-300amu, with a minimum scan cycle of 1 scan per second

10.1.2 Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

10.1.2.1 "Three ring trap"-charcoal, Tenax, silica gel
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 180C
Bake time: 8 minutes at 225C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

10.1.2.1 VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40C
Desorb time: 4 minutes
Desorb temperature: 225C
Bake time: 8 minutes at 250C
Purge flow: Approximately 20-30mL/minute
Valve temperature: 100C
Transfer line: 100C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets-ketones, ethers, bromoform-may be low and the reporting limit may not be achieved on a routine basis.

10.2 BFB Tune Check

10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the "**tune**" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

10.2.2 Evaluation of the 4-BFB peak.

10.2.2.1 The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans +/- 5 scans from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

10.2.2.3 The following records must be kept for each 4-BFB analysis that meets the criteria:
- the date, time, and data file of the analysis
- a spectrum of the scan or averaged scans
- a tabulation of the ion abundances of the scan

10.2.2.4 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open , or other anomalies.

10.2.2.5 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

10.3 Initial Calibration

After the 4-BFB criteria has been met, the initial calibration standards are analyzed. Prepare the initial calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

10.3.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water (syringe valve in the "red" position).

10.3.2 Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL or 25mL)

10.3.3 Briefly remove the syringe valve and inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.3.4 Load the standard(s) onto the purge and trap device and begin the analysis. All pertinent information concerning the standards must be recorded on the analysis log. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

10.3.5 After the acquisition has taken place, evaluate the calibration standards to ensure that each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.

10.3.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system or using a PC spreadsheet as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where
Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug on-column)
Cis = concentration or mass on-column of the internal standard (ug/L or ug/kg OR ng or ug on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound:

$$RRFavg = \frac{RRF1 + RRF2 + ... + RRFn}{n}$$

where n = number of calibration levels

Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - RFavg)^2}{n-1}}$$

where
Rfi = response factor of a target compound in the individual calibration level
Rfavg = average response factor
n= number of calibration levels

10.3.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target:

$$\%\,RSD = \frac{standard\ deviation}{RRFavg} \otimes 100$$

10.3.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

Calibration Check Compounds – CCC Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.3.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration or mass on-column of the target compound being measured (ug/L or ug/kg OR ng or ug)
Cis = concentration of the internal standard (ug/L or ug/kg OR ng or ug)

If the correlation coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples.. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3.10 After the initial calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.

If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

10.4 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. This criteria must be met before the analysis of the calibration check standards can take place.

10.4.1 After the tune criteria has been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a nominal concentration of 50ug/L-kg for 5ml/5g samples and 10ug/L for 25mL with ketones and poor purgeables at higher concentrations. The CCC and SPCC criteria (Section 10.3.8) must be met before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration
Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If this criteria is not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.4.2 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.4.3 After the continuing calibration criteria has been met, the method blank is analyzed. 5.0mL or 25mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in Table 5 of the STL-SL CQAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compound must meet the criteria.

If the concentration of any target compound is above the RL in Table 5 of the STL-SL CQAP, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.5 Aqueous Sample Analysis-5.0mL to 25mL

The analyst must use the same volume as was used for the calibration standards-if a 5mL sample is used, it must be quanted off of the 5mL calibration curve; if a 25ml sample is used, it must be quanted off of the 25mL calibration curve. Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria has been met, and the method blank criteria has been met. See the SOP Summary for the analytical sequence.

10.5.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.

10.5.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. ***Gloves must be worn when handling samples.***

10.5.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.

10.5.5 Remove the plunger from the glass syringe. Attach a syringe valve to the syringe Luer-tip to prevent sample from spilling out of the syringe when sample is added.

10.5.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.

10.5.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve , and expel the air while adjusting the volume to 5.0mL or 25mL. If no air bubbles were trapped, adjust the syringe to volume.

NOTE: For TCLP leachate samples, use 1.25mL of sample (1:4 dilution).

10.5.7 Open the syringe valve and inject the internal standard/surrogate (ISSU) mix into the sample.

10.5.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information on the analysis log. Check the pH of the sample with pH paper and record the pH on the instrument log or other appropriate log.

10.5.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.

10.5.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL or 25ml,depending on which curve is being used. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve.

$$DF = \frac{final\ volume\ of\ dilution(mL)}{volume\ of\ sample\ used(mL)}$$

For example, if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5). If 1.0mL of sample is diluted to a final volume of 25mL, the dilution factor is 25 (25/1=25).

The following table gives some dilution factors:

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50
25.0	0	25.0	1
5.0	20.0	25.0	5
2.5	22.5	25.0	10
1.0	24.0	25.0	25
0.50	24.5	25.0	50
0.10	24.9	25.0	250

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.6 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5mL purge. The tune, initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the sample to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

Liquid field QC for soils (trip blank, field blank, etc.) should be analyzed with the associated soil samples, using the same preparation and analytical procedures, including the heated purge. Report the results for liquid trip blanks as ug/L.

10.7 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000ug/kg). or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria has been met. Medium level soil extracts are quanted using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

10.7.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water(syringe valve in the "red" position). Replace the plunger, switch the syringe valve to "green", and force any airspace out of the syringe. Adjust the volume to the syringe volume(5mL)

10.7.2 Briefly remove the syringe valve and inject the sample extract and 5uL of the internal standard (IST) solution into the syringe. Use 125ul of the extract for soils and 100uL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceed the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (Section 9).

10.7.3 Load the sample on to the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded on the analysis log. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as was used for the initial and continuing calibration standards-ambient purge for aqueous samples.

10.7.4 Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is 0.0025mL (2.5uL).

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates- Theoretical ng on-column
125uL(0.125mL)	100uL (0.100mL)	250
62.5uL(0.0625mL)	50uL(0.050mL)	125
25uL(0.025mL)	25uL(0.020mL)	50
12.5uL(0.0125mL)	10ul(0.010mL)	25
2.5uL(0.0025mL)	2.0uL(0.0020mL)	5.0-quantiation limit
<2.5uL(0.025mL)	<2.0uL(0.0020mL)	<5.0ng- below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification. Tentative identifications of non-targets will be made only by analysts having completed the training specified in the training schedule.

11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree within +/-30%.

11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult other analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where
Cis = concentration of the internal standard, ug/L
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
DF = dilution factor

Soils by Heated P/T

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
W = weight of sample analyzed, g
solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC\,(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where
Cis = concentration of the internal standard, ug/kg
AREAis = total ion peak area of the internal standard
AREAtic= total ion peak area of the TIC
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = decimal equivalent of the percent solids(percent solids/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where
Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples-Internal Standard Technique

Aqueous Samples- relative response factor :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
DF = dilution factor

Aqueous Samples: regression curve

$$concentration(ug/L) = concentration(curve) \otimes DF$$

where
DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(ug/L) = RLqap \otimes DF$$

where
DF = dilution factor. The SL CQAP Table 5 RL(RLqap) assumes a DF of 1.

Soils by Heated P/T- relative response factor :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/kg)
RRFavg = average response factor of the compound being measured
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

Soils by Heated P/T: regression curve

$$conc(ug/kg,dw) = Ccurve(ug/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where
Ccurve = concentration from curve(ug/kg)
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The reporting limit (RL) is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the sparging vessel (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes- relative response factor

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$$

where
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (ug/L)
RRFavg = average response factor of the compound being measured
Vcal = volume that calibration curve is based on (5mL or 25mL)
solids = (percent solids)/100)
W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g)
Vf = final volume of the extract (mL)
Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(ug,kg,dw) = Ccurve(ug/L) \otimes \frac{Vcal}{(W)(solids)}$$

where
Vcal = volume that calibration curve is based on (0.005L or 0.025L)
W = weight of sample added to the reagent water (g)-defined above

The reporting limit (RL)is calculated:

$$RL = RLqap \otimes \frac{5.0g}{(W)(solids)}$$

where
W = weight of sample added to the reagent water (g)
solids = (percent solids)/100)

The STL-SL CQAP assumes W= 5.0g and solids = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in Section 3.1.3 of STL-SL SOP AN02: *Analytical Batching*. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.

STL-SLSOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. This criteria is summarized in the attached 8260 SOP Summary.

STL-SL SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must demonstrate competence in the analysis of samples by this procedure. The minimum criteria for this demonstration is the preparation and analysis of spiked reagent water. Section 8.3 of EPA Method 8260A gives the general procedure for the performance of the IDOC and Table 6 of EPA Method 8260A gives the acceptance criteria for the accuracy and precision.

12.3 Method Detection Limit

The method detection limit is determined in accordance with STL-SL SOP CA90.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance items will be added at a later date. Section 10 of the STL-SL QAPs provide guidance on preventive maintenance.

14.0 TROUBLE-SHOOTING

Trouble-shooting items will be added at a later time. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

15.0 REFERENCES

1. *Savannah Laboratories' Comprehensive Quality Assurance Plan* and *Savannah Laboratories' Corporate Quality Assurance Plan*, current revisions.
2. Method s 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846.including Update III* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

Appendix A

VOLATILES BY GC/MS WORKING STANDARDS -EXAMPLE

These standards can be used to prepare the working standards for EPA Method 8260 to report the TCL (target compound list) compounds and the extended list of target compounds generally associated with EPA 8260. The standards are prepared in purge and trap grade methanol and are stored at 4C with minimum headspace.

Working Standard 1 (TCL WS-1)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #2	2000	12.5	25
VOA Cal #3	2000	12.5	25
VOA Cal #4	2000	12.5	25
1,2,-DCB	5000	5.0	25
1,3-DCB	5000	5.0	25
1,4-DCB	5000	5.0	25
2-CEVE	1000	125	125

Working Standard 2 (TCL WS-2)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA Cal #1	5000	25	125
8260 Surrogates	2500	10	25

Working Standard for GASES (TCL GASES)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
502.2 Cal 1	2000	12.5	25

Appendix A

Working Standard 3 (8260 WS-3)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
8260 Custom Mix #1	200	125	25
8260 Custom Mix #2	200	125	25
1,1,2,2-Tetrachloroethane	2000	12.5	25

Appendix A

Internal Standard (8260 ISTD)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50

Internal Standard/Surrogate (8260 ISSU)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC (ug/mL)
VOA ISTD	2500	20	50
1,2-DCE-d4	2000	25	50
8260 Surrogate	2500	20	50

Tune Evaluation Standard (4-BFB)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
4-BFB	5000	10	50

Matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
Matrix Spiking Solution	2500	20	50

TCLP matrix Spike Standard (5-component subset)

STOCK STANDARD	CONC (ug/mL)	microliters of stock to final volume of 1.0mL	STD CONC. ug/mL
TCLP Spiking Solution	2000	16	125

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS - EXAMPLES

The following calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	TCL-1 *	TCL-2 *	TCL-3 *	TCL-4 *	TCL-5 *	TCL-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	8260-1 *	8260-2 *	8260-3 *	8260-4 *	8260-5 *	8260-6 *
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 5.0mL of reagent water or to 5.0g of blank sand.

CONCENTRATIONS OF THE CALIBRATION STANDARDS-5.0mL OR 5.0g

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
TCL-1,8260-1	5ug/l-kg	25ug/l-kg
TCL-2,8260-2	10ug/l-kg	50ug/l-kg
TCL-3,8260-3	25ug/l-kg	125ug/l-kg
TCL-4,8260-4	50ug/l-kg	250ug/l-kg
TCL-5,8260-5	100ug/l-kg	500ug/l-kg
TCL-6,8260-6	200ug/l-kg	1000ug/l-kg

Appendix A

VOLATILES BY GC/MS CALIBRATION STANDARDS-25mL Purge Volume-EXAMPLES

These calibration standards are prepared to define the working range of the EPA 8260 analysis for the target compound list (TCL) and the extended list of compounds generally associated with EPA 8260. The standards are based on a volume of 25mL to achieve lower quantitation limits for the target compounds. The lowest level standard is at the reporting limit and the other standards define the working range. Samples with target analytes above the concentration of the highest calibration standard must be diluted and reanalyzed.

TARGET COMPOUND LIST

Working Level standards	Conc (ug/mL)	25TCL -1*	25TCL-2*	25TCL-3*	25TCL-4*	25TCL-5*	25TCL-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

8260 EXTENDED LIST (TCL+ADDITIONAL COMPOUNDS)

Working Level standards	Conc (ug/mL)	258260-1*	258260 -2*	258260-3*	258260-4*	258260-5*	258260-6*
TCL WS-1	25/125	1.0	2.0	5.0	10.0	20.	40
TCL WS-2	125	1.0	2.0	5.0	10	20	40
8260 WS-3	25	1.0	2.0	5.0	10	20	40
TCL GASES	25	1.0	2.0	5.0	10	20	40
TCL ISTD	50	5.0	5.0	5.0	5.0	5.0	5.0

*uL of the working standard added to 25mL of reagent water.

CONCENTRATIONS OF THE CALIBRATION STANDARDS

Cal Std	all targets except ketones, 2-CEVE	ketones, 2-CEVE
25TCL-1,25-8260-1	1.0ug/l	5.0ug/l
25TCL-2,25-8260-2	2.0ug/l	10ug/l
25TCL-3,25-8260-3	5.0ug/l	25ug/l
25TCL-4,25-8260-4	10ug/l	50ug/l
25TCL-5,25-8260-5	20ug/l	100ug/l
25TCL-6,25-8260-6	40ug/l	200ug/l

Appendix B
8260 SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Container	Hold Time
Aqueous	None; 4C	40mL no headspace	7 days
	HCl pH<2; 4C	40mL-no headspace	14 days
Soil/solid(low level)	Iced at collection; 5mL sodium bisulfate added upon arrival in lab; store at 4C	5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab; store at –10C	5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4C	Glass 125mL	14 days
TCLP	HCl pH<2; 4C	Tedlar bag or syringe	14 days

*storage temperature is 4C with a control criteria of less than 6C with no frozen samples

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column Clock starts at injection	4-BFB 50ng on column Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification (50ug/L or 50ug/kg)) RL Standard-low point on cal curve (if necessary)
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

See SL SOP AN02, Section 3.1.3, for the batch/clock options for LCS and MS/MSD.

Recommended Internal Standards:
1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

Surrogates/System Monitoring Compounds:
dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS: CQAP Subset:
1,1-dichloroethene; benzene; trichloroethene; toluene; chlorobenzene

Appendix B
8260 SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)	
m/e	**Abundance Criteria**
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

(1) *8260 criteria taken from CLP OLMO4.0 (January 1998)

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	**Continuing Calibration**
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

System Performance Check Compounds-SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30 (0.10 for 25mL purge volume)

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check - 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see -Calibration Acceptance Criteria – Table presented earlier in this document)	- Evaluate chromatograms, spectra, and integrations - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument maintenance and recalibrate
Continuing Calibration check - midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria - Table presented earlier in this document)	- Evaluate chromatogram, spectra, integrations - Reanalyze standard - Remake and reanalyze standard - Recalibrate - Perform instrument maintenance and recalibrate
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <RL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Lab Control Sample (LCS) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Matrix Spike/Matrix Spike Duplicate (MS/MSD) -subset of target compounds unless full target spike specified by client	Each batch	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogates	All samples, blanks, LCS, MS	STL-SL CQAP Section 5	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in STL-SL SOP AN02 and Table 13.1 in CQAP -Perform instrument or column maintenance, recalibrate, and reanalyze
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence -Retention time of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be evaluated for gross error . Consult supervisor.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze

Appendix B

QC Check	Frequency	Acceptance Criteria	Corrective Action
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate
Initial Demonstration of Capability	Per analyst	Method criteria	-Reanalyze targets that do not meet criteria
Method Detection Limit (MDL)	See STL-SL SOP CA90	See STL-SL SOP CA90	-Reanalyze and re-evaluate

Appendix C
EXAMPLE QUANTITATION REPORT

-quantitation ions
-internal standard and target compound association

SEMI-VOLATILE COMPOUNDS BY GC/MS
Method: 8270C

Approved by:

R. Wayne Robbins — 30 Aug 2002
Date

Title: Technical Manager

STL X Savannah ___ Tallahassee ___ Mobile ___ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of various semi-volatile organic compounds (SVOC) in groundwater, TCLP and SPLP leachates, soils, sediments, wastes, and solid sample extracts. The attached quantitation report (Appendix B) lists the routine target compounds, the retention times of the target compounds, the characteristic ions of the target compounds, and the internal standard associated with each target compound.

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits for the target compounds are given in Section 5 of the current revision of the Laboratory Quality Manual (LQM).

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0mL, and analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

2.2 This procedure is based on the guidance provided in SW-846 Method 8270C.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 The toxicity or carcinogenicity of each chemical used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves, and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated area.

3.3 Material Safety Data Sheets (MSDS) are available to the analyst. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

3.4 The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of the extract. The traps should be changed every six months and disposed of in accordance with SOP CA70: *Waste Management.*

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Glassware and/or extraction vessels that have not been properly cleaned may contribute artifacts that make identification and quantification of the target compounds difficult. Elevated baselines may be due to oils, greases, or other hydrocarbons that may be extracted from improperly cleaned glassware or extraction vessels.

4.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. Sample extracts that contain high concentrations of non-volatile material such as lipids and high molecular weight resins and polymers may require the optional GPC cleanup prior to analysis. The GPC cleanup is generally not effective in removing non-target material that is associated with common petroleum products like diesel.

4.3 Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days from TCLP leaching procedure	40 days

Refrigerator temperature acceptance criterion is less than 6C with no frozen samples.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph- Hewlett-Packard (HP) 5890 or equivalent with compatible autosampler, splitless injector, and direct capillary interface. The exit vent of the splitless injector must have a carbon trap in-line to collect the semivolatile compounds that are vented during the injection of extracts. The carbon traps should be changed every six months.

6.2 Mass spectrometer- HP5971, HP5972, HP5973 or equivalent

6.3 Recommended Capillary column-HP-5MS, 30m x 0.25mm ID x 0.25um film thickness or equivalent column

6.4 Data system- compatible with GC/MS system

6.5 Microsyringes- appropriate volumes

6.6 Volumetric flasks- Class A, appropriate volumes

6.7 Autosampler vials and crimper- compatible with autosampler

7.0 REAGENTS

Reagents must be tracked in accordance with SOP AN44: *Reagent Traceability.*

7.1 Methylene chloride- pesticide residue grade, for preparation of standards

7.2 Acetone- pesticide residue grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation from neat materials.

8.1 Preparation of the Stocks from Neat Standards

The steps for the preparation of primary stock standards from neat materials are given in SOP *AN43: Standard Preparation.* The standards should be prepared in methylene chloride but may require other solvents to dissolve the material.

8.2 Preparation the calibration standards from the stock standards

A minimum of five calibration standards are prepared. The concentrations of the stock standards are in the 1000-10000ug/mL range. The recommended standards are listed in Section 10.2. The lowest level standard should be at the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve. Internal standards should be added to each standard to give a final concentration of 40ug/mL.

Each lab should develop controlled recipes that can be posted or maintained in appropriate logbooks.

9.0 SAMPLE PREPARATION

9.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP	Extraction Technique
Aqueous, TCLP leachates	EX30	Continuous Liquid-liquid Extraction
Aqueous, TCLP leachates	EX35	Separatory Funnel
Soils/Sediments	EX40	Sonication
Wastes	EX42	Waste dilution

9.2 The sample concentration procedures are given in SOP EX 50: Zymark Nitrogen Concentration.

9.3 Gel permeation chromatography (SOP EX61) may help to eliminate or minimize matrix interferences in a limited number of samples. The GPC cleanup is generally not effective on samples containing petroleum products.

10.0 PROCEDURE

10.1 Instrument Conditions

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The lab must optimize and document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

HP-5MS 30m x 0.25mm ID x 0.25um film thickness or equivalent

Column flow: Approximately 1mL/min helium

GC Oven temperatures:

Initial column temperature: 45 C for 3 minutes
Column temperature program: 10C per minute
Final column temperature: 300C (until at least one minute past the elution time of Benzo (g,h,i) perylene).

GC injector parameters

Injector temperature: 250-270EC
Injection type: split, approximately 1:10 or splitless injection
Injector liner: 4mm ID quartz or 4mm glass, deactivated (single "Gooseneck")
Sample injection volume: 1-2uL

Mass Spectrometer and interface parameters

Mass spectrometer interface: 300C
Mass spectrometer source temperature: Factory Set
Mass range: 35-500amu, with a scan time of 1.0 scans per second or greater

10.2 Calibration

A minimum of five calibration standards are prepared and analyzed. The recommended standards are 10, 20, 50, 80, 100, 200ug/mL. The lowest level standard should be at or below the equivalent of the reporting limit and the rest of the standards should define the working range of the detector. Note that six calibration levels are required for a second order regression curve.

10.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions must be identical-the mass range, scan rate, and multiplier voltage.

10.2.1.1 Prepare a 50ng/uL solution of tune/column evaluation standard containing each of the following compounds at 50ug/mL in methylene chloride: DFTPP, pentachlorophenol, p,p'-DDT, and benzidine.

10.2.1.2 Analyze a 1uL aliquot of the tune/column evaluation solution.

10.2.1.3Evaluate the DFTPP peak.

-The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

-The spectrum of the DFTPP must meet the criteria listed in the SOP Summary (Appendix A). Background subtraction must be straightforward, that is, no scan within the elution window of DFTPP may be subtracted from another scan within the elution window, and designed only to eliminate column bleed or instrumental background. Scans +/- 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

NOTE: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.1.4Benzidine and pentachlorophenol should be present at their normal responses with minimal peak tailing visible. Peak tailing guidance is taken from EPA Method 625 which allows pentachlorophenol to be less than or equal to five and benzidine less than or equal to three. Refer to Figure 1for an example of [eak tailing factor calculation.

This is a good check on the system: if pentachlorophenol (a CCC) does not respond well, the calibration standard should not be analyzed. Injector port and column maintenance should be performed and the tune/column evaluation standard reanalyzed.

The percent breakdown of p,p'- DDT is calculated using the following equation. The percent breakdown should not exceed 20%.

$$\%Breakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\ 100$$

Areas from the total ion chromatogram are used to calculate DDT breakdown.

10.2.2 After the DFTPP criteria and column evaluation criteria have been met, the initial calibration standards are analyzed.

10.2.2.1Prepare the initial calibration standards. The lowest calibration standard should be at the RL and the rest of the standards will define the working range. See section 10.2 for guidance regarding calibration levels.

10.2.2.2Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts.

10.2.3 Identify the internal standards, surrogates, and the target compounds. The data system must be updated with the proper retention times and ion data.

10.2.4 Calculate the relative response factor for each compound as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured
(See the attached quantitation report for a list of the compounds that are associated with the correct internal standard)
Cx = concentration of the compound being measured (ug/mL)
Cis = concentration of the internal standard (40ug/mL)

Secondary ions may be used for quantification if there is interference with the primary quantitation ion. If a secondary ion is used for quantification, the concentration/response relationship of the secondary ion must be established. The secondary ion must meet the same calibration criteria as the primary ion.

10.2.5 Calculate the average relative response factor (RRF_{avg}) for each target compound and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3.... + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

10.2.6 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\frac{\sum_{i-1}^{n_1} (RRF_i - RRF_{avg})^2}{n-1}}$$

10.2.7 Calculate the relative standard deviation (%RSD) of the target compounds in the calibration standards.

$$\%RSD = \frac{SD}{RRF_{avg}} x\,100$$

10.2.8 Evaluation of the Initial Calibration

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in the SOP Summary (Appendix A). The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of sample extracts can begin.

If the CCC and SPCC criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.2.9 After the initial calibration criteria (CCC/SPCC) have been met, each target is evaluated for linearity. Refer to SOP AN67: *Evaluation of Calibration Curves* for guidance.

If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples.

If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios-Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where:

Ax = area of the characteristic ion for the compound being measured
Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds and their associated internal standard)
Cx = concentration of the target compound being measured (ug/mL)
Cis = concentration of the internal standard (ug/mL)

A linear or quadratic curve may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire concentration range.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of the calibration curve be eliminated without eliminating the entire level.

8000B exception: evaluation of the "grand mean": If the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

10.3 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of the tune/column evaluation solution (10.2.1.1). The tune and column evaluation criteria (10.2.1.3 and 10.2.1.4) must be met before the analysis of the calibration check standards can take place.

10.3.1 After the tune and column evaluation criteria have been met, a continuing calibration check standard(s) is analyzed. The continuing calibration standard should be at a mid-level concentration. The CCC and SPCC criteria (SOP Summary, Appendix A) must be met before the analysis of samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where
RRFavg = average response factor from initial calibration
RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where
Ci = Calibration Check Compound standard concentration (ug/mL)
Cccv = measured concentration using the selected quantitation method (ug/mL)

NOTE: The SPCC criteria (10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

10.3.2 The continuing calibration verification standard (CCV) must also be evaluated for internal standard response.

If the extracted ion current profile (EICP) area for any of the internal standards in the CCV changes by more than a factor of two (-50% to +100%) from the last initial calibration sequence, the analytical system must be inspected for problems and corrective action instituted.

10.4 Samples are analyzed only after the DFTPP criteria, column evaluation criteria and the calibration verification criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the tune/column evaluation standard and a mid-level calibration standard.

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- Minimum of five cal levels	Mid point calibration verification Optional RL: Standard-low point on cal curve
Samples analyzed until 12-hour clock expires	Samples analyzed until 12-hour clock expires

10.4.1 Remove the sample extracts to be analyzed from the refrigerator and allow the sample to come to ambient temperature.

10.4.2 Add 20-uL of the internal standard mix (2000 ug/mL) to each 1.0mL aliquot of the sample extract. The concentration of the internal standard in the extract is 40 g/mL.

10.4.3 Mix the contents of the autosampler vial by inverting several times.

10.4.4 Analyze the samples using the same analytical conditions used for the initial and continuing calibration standard. Determine the concentration of the samples and QC items using the procedures of Section 11. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level with out instrument overload.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with t the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

10.4.5 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100uL of a sample extract are diluted to final volume of 1.0mL, the dilution factor is 10. (1000/100 = 10). The following table gives some dilution factors:

Dilution Preparation

uL extract-Vext	uL MeCl2	volume of dilution (Vdil-uL)	uL ISTD (2000ug/mL)-Vistd	DF
1000	0	1000	20	1
500	500	1000	10*	2
200	800	1000	16*	5
100	900	1000	18*	10
50	950	1000	19*	20
20	980	1000	20*	50

*assumes dilution of a 1mL extract or 1mL aliquot of an extract that has been spiked with the internal standard at 40ug/mL using 20ul of a 2000ug/mL internal standard solution

The concentration of internal standards must remain constant for all extracts and extract dilutions at 40ug/mL. The following equation can be used to determine the volume of the 2000ug/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$Vistd(uL) = 20uL - \left(\frac{Vext}{Vdil} \otimes 20ul\right)$$

Vistd = volume of 2000ug/mL internal standard to add to the diluted extract (uL)
Vext = volume of extract used to prepare the dilution (uL)
Vdil = final volume of the dilution (uL)-1000uL (1.0mL)

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Qualitative Analysis

11.1.1 Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the daily calibration standard or a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to positively identify a compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Tentatively Identified Compounds (TICs)

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification.

The default procedure is to evaluate up to 20 compounds of greatest apparent concentration that are not included as target compounds or routinely reported volatile compounds. The unknown compounds are tentatively identified using a forward search of the reference library.

If the library search produces a match at or above 85%, report that compound. If the library search produces more than one compound at or above 85%, report the first compound (the highest match quality). If the library search produces no matches at or above 85%, report the compound as unknown. If possible, provide a general classification of the unknown – for example, unknown aromatic, unknown hydrocarbon, etc.

TICs should be evaluated within the retention time range from the first eluting target or surrogate (whichever is first in the target list) to three minutes after the elution of the last target compound.

11.1.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.2.2 The relative intensities of the major ions should agree within +/-20%.

11.1.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.

11.1.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

11.1.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.

11.1.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria is not met exactly, the peak may be considered tentatively identified. The analyst should consult senior analysts or the mass spectral interpretation specialist if there are any questions concerning an interpretation of spectra.

11.1.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculations assume that the same volume is injected for standards and samples.

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{V} \otimes DF$$

where:

Cis =	concentration of the internal standard (ug/mL)
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract (mL)
V =	volume of sample extract (L)
DF =	dilution factor

Soils

$$TIC\ (ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{F}{(W)(solids)} \otimes DF$$

where:

Cis =	concentration of the internal standard, ug/mL
AREAis =	total ion peak area of the internal standard
AREAtic=	total ion peak area of the TIC
F =	final volume of extract mL
W =	weight of sample analyzed (kg)
solids =	decimal equivalent of percent solids

11.2 Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples and that the standards and samples have the same concentration of internal standard.

11.2.1 Aqueous Samples

11.2.1.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{V} \otimes DF$$

where:

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.2 If a regression curve is used, the concentration is given:

$$concentration(ug/L) = Ccurve \otimes \frac{F}{V} \otimes DF$$

where:

C_{curve} =	concentration from curve (ug/mL)
F =	final volume of extract (mL)
V =	volume of sample extracted (L)
DF =	dilution factor

11.2.1.3 The reporting limit (RL) for each sample is given:

$$RL(ug/L) = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Vqap}{V} \otimes DF$$

where:

F = final volume of extract (mL)
Fqap = 1.0mL
Vqap = 1.0L
V = volume of sample extracted
DF = dilution factor. The LQM RL assumes a DF of 1.

NOTE: If V = 800mL to 1200mL, assume that Vqap/ V = 1 in the calculation of the reporting limit.

11.2.2 Soils

11.2.2.1 If the relative response factor is used, the calculation for samples is :

$$concentration(ug/kg,dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ax =	area of the characteristic ion of the compound being measured
Ais =	area of the characteristic ion of the internal standard
Cis =	concentration of the internal standard (ug/mL)
RRFavg =	average response factor of the compound being measured
F =	final volume of extract (mL)
W =	weight of sample extracted (kg)
solids =	(percent solids)/100
DF =	dilution factor

11.2.2.2 If the regression curve is used, the concentration is given:

$$conc(ug/kg,dw) = Ccurve \otimes \frac{F}{(W)(solids)} \otimes DF$$

where

Ccurve =	concentration from curve(ug/mL)
W =	weight of sample extracted (kg)
F =	final volume of extract (mL)
solids =	(percent solids)/100)
DF =	dilution factor

11.2.2.3 The reporting limit (RL) for each sample is given:

$$RL = RLqap \otimes \frac{F}{Fqap} \otimes \frac{Wqap}{(W)(solids)} \otimes DF$$

where

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100

The LQM assumes Wqap = 30g, solids = 1, Fqap = 1.0mL, and DF = 1.

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in AN02: *Analytical Batching.* SOP AN02 also describes the procedure for evaluating batch-specific QC. The QA/QC criteria are summarized in the SOP Summary (Appendix A).

12.2 Initial Demonstration of Capability (IDOC) to Generate Acceptable Accuracy and Precision

Each analyst must participate in the analysis of samples by this procedure in accordance with SOP CA92: *Evaluation of IDOCs.*

12.3 Method Detection Limit

The method detection limit is determined in accordance with SOP CA90: *Procedure for the Determination of the Method Detection Limit.*

13.0 PREVENTIVE MAINTENANCE & TROUBLESHOOTING

Refer to SOP AN53: *Preventive Maintenance Procedures for Laboratory Instruments* for guidance.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Refer to SOP CA70: *Waste Management* for proper waste handling procedures.

15.0 REFERENCES

15.1 *STL Savannah Laboratory Quality Manual* current revision.

15.2 Method 8270C:*Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

APPENDIX A
8270C SOP SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	none; 4C	1-L amber	7 days	40 days
Soil/ Sediment	none; 4C	500-mL	14 days	40 days
Waste	none; 4C	Glass	14 days	40 days
TCLP	none; 4C	1-L amber	7 days	40 days

ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
Tune/Column Evaluation Standard Clock starts at injection	Tune/Column Evaluation Standard Clock starts at injection
Calibration standards- minimum of five cal levels	Mid point calibration verification standard RL Standard (lowest point on calibration curve if required by client or state-specific QAP)
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)	
m/e	**Ion Abundance Criteria (1)**
51	30-80% of mass 442
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	25-75% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10-30% of mass 198
365	Greater than 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15.0-24.0% of mass 442

(1) 8270 criteria taken from CLP OLMO4.0 (January 1998). The use of alternate criteria is expressly allowed in SW-846 Method 8270C.

APPENDIX A
8270C SOP SUMMARY

CALIBRATION ACCEPTANCE CRITERIA

Calibration Check Compounds - CCC
Phenol, 1,4-Dichlorobenzene, 2-Nitrophenol, 2,4-Dichlorophenol, Hexachlorobutadiene, 4-Chloro-3-methylphenol, 2,4,6-Trichlorophenol, Acenapthene, N-Nitrosodiphenylamine, Pentachlorophenol, Fluoranthene, Di-n-octylphthalate, Benzo(a) pyrene

System Performance Check Compounds-SPCC
N-Nitrosodi-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol

Initial Calibration	Continuing Calibration*
CCC: <= 30% RSD SPCC: RRFavg >= 0.050	CCC: <= 20% difference from initial calibration SPCC: RRF>= 0.050

*If CCC and/or SPCC do not meet the stated criteria, all targets that are reported must meet the CCC criteria.

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

The results for all target compounds are evaluated for linearity. If the %RSD is less than 15%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation. If the average response factor for the target exceeds 15% (including any CCC), the analyst must use the calibration curve option.

NOTE: The lab has the option of using a regression curve for all analytes.

A linear, quadratic, or higher order regression fit may be used to define the concentration/response relationship. If r^2 is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. The minimum number of calibration standards required for a regression curve are given in the following table:

Type of curve	Minimum Number of Calibration Points
Linear (first order)	5
Quadratic (second order)	6

QC Item	Frequency	Acceptance Criteria	Corrective Action
Tune/Column Evaluation Standard DFTPP 50ng Pentachlorophenol - 50ng Benzidine – 50ng p,p'-DDT 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Evaluate alternative scans -Reanalyze and evaluate -Retune and reanalyze -Clean source, retune, reanalyze
		Pentachlorophenol and benzidine - present at usual response with no peak tailing visible p,p'-DDT - %breakdown <20%	-Reanalyze -Perform injector port maintenance and reanalyze -Cut more than usual length of column and reanalyze -Replace column
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards	CCC: %RSD < 30% SPCC: RRFavg > 0.050 Use regression curve for quantitation if %RSD for any target compound exceeds 15%	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	CCC: %Difference <= 20% Or %Drift <= 20% SPCC: RRF >= 0.050	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous initial calibration sequence Areas in samples should be evaluated for gross error. Consult supervisor Retention time of internal standard must be +/-30 seconds from internal standard in previous CCV.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available -Recalibrate

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >RL	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets < RL in LQM	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Lab Control Standard (LCS) - QAP subset	Per batch See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
Matrix spike (MS) Matrix spike duplicate (MSD)	Per batch if sufficient sample volume/weight supplied See SOP AN02	Within LQM Control Limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Follow guidance in STL-SL SOP AN02
RL Standard (reporting limit)	Daily (optional)-lowest point on calibration curve if required by client or state-specific QAP	Detected at reasonable sensitivity	-Evaluate integrations and spectra; -Reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Each work group	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria
Method Detection Limit (MDL)	Annually for each routine matrix See SOP CA90	Evaluate according to SOP CA90	Evaluate according to SOP CA90

APPENDIX B- TARGET COMPOUNDS

ROUTINE TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions		ISTD
1,4-Dioxane	1.894	88	58	45	1
Pyridine	2.123	79	52	51	1
N-Nitrosodimethylamine	2.102	42	74		1
Aniline	3.812	93	66		1
Phenol	3.796	94	66	65	1
Bis(2-chloroethyl)ether	3.854	63	93	95	1
2-Chlorophenol	3.908	128	130	64	1
1,3-Dichlorobenzene	4.025	146	148	111	1
1,4-Dichlorobenzene	4.073	146	148	111	1
Benzyl Alcohol	4.202	108	79	77	1
1,2-Dichlorobenzene	4.239	146	148		1
2-Methylphenol	4.314	107	108	77	1
bis(2-Chloroisopropyl)ether	4.335	45	121		1
N-Nitroso-di-n-propylamine	4.469	70	42	101	1
3&4-Methylphenol	4.447	107	108		1
Hexachloroethane	4.522	117	201	199	1
Nitrobenzene	4.602	77	123	65	2
Isophorone	4.837	82	95	138	2
2-Nitrophenol	4.923	139	109	65	2
2,4-Dimethylphenol	4.965	107	122	121	2
Bis(2-chloroethoxy)methane	5.067	93	123	95	2
Benzoic acid	5.115	105	122		2
2,4-Dichlorophenol	5.169	162	164	98	2
1,2,4-Trichlorobenzene	5.259	180	182	145	2
Naphthalene	5.323	128	129		2
4-Chloroaniline	5.409	127	129	65	2
Hexachlorobutadiene	5.532	225	223	227	2
4-Chloro-3-methylphenol	5.991	107	144	142	2
2-Methylnaphthalene	6.135	142	141		2
1-Methylnaphthalene	6.269	142	141		2
Hexachlorocyclopentadiene	6.429	237	235	272	3
2,4,6-Trichlorophenol	6.541	196	198	200	3
2,4,5-Trichlorophenol	6.590	196	198	200	3
2-Chloronaphthalene	6.760	162	164	127	3
2-Nitroaniline	6.958	65	92	138	3
Dimethylphthalate	7.268	163	194	164	3
2,6-Dinitrotoluene	7.353	165	89	63	3
Acenaphthylene	7.337	152	151	153	3
3-Nitroaniline	7.540	138	108	92	3
Acenaphthene	7.599	154	153	152	3
2,4-Dinitrophenol	7.685	184	63	154	3
4-Nitrophenol	7.824	65	109	139	3
Dibenzofuran	7.829	168	139		3
2,4-Dinitrotoluene	7.914	165	89	63	3
2,3,4,5-Tetrachlorophenol	8.064	232	230	131	3
2,3,4,6-Tetrachlorophenol	8.091	232	230	131	3
Diethylphthalate	8.310	149	177	150	3

Fluorene	8.336	166	165	167	3
4-Chlorophenyl-phenylether	8.363	204	141	206	3
4-Nitroaniline	8.454	138	108	92	3
4,6-Dinitro-2-methylphenol	8.513	198	105	121	4
N-Nitrosodiphenylamine	8.555	169	168	167	4
1,2-Diphenylhydrazine	8.593	77	105	182	4
4-Bromophenyl-phenylether	9.090	248	250	141	4
Hexachlorobenzene	9.293	284	142	249	4
Pentachlorophenol	9.581	266	264	268	4
Phenanthrene	9.784	178	176	179	4
Anthracene	9.854	178	176	179	4
Carbazole	10.137	167			4
Di-n-Butylphthalate	10.847	149	150	104	4
Fluoranthene	11.659	202	203	101	4
Benzidine	11.926	184	92	185	5
Pyrene	12.006	202	200	203	5
Butylbenzylphthalate	13.214	149	91	206	5
3,3'-Dichlorobenzidine	13.892	252	254	126	5
Benzo(a)Anthracene	13.866	228	229	226	5
Bis(2-ethylhexyl)phthalate	14.111	149	167	279	5
Chrysene	13.924	228	226	229	5
Di-n-octylphthalate	14.971	149	43		5
Benzo(b)fluoranthene	15.367	252	253	125	6
Benzo(k)fluoranthene	15.399	252	253	125	6
Benzo(a)pyrene	15.783	252	125	253	6
Indeno(1,2,3-cd)pyrene	17.284	276	139		6
Dibenzo(a,h)anthracene	17.317	278	139	279	6
Benzo(g,h,i)perylene	17.674	276	277	138	6
SURROGATES					
2-Fluorophenol	3.032	112	64		1
Phenol-d5	3.785	99	71		1
Nitrobenzene-d5	4.586	82	128	54	2
2-Fluorobiphenyl	6.643	172	171		3
2,4,6-Tribromophenol	8.732	330	332	141	3
Terphenyl-d14	12.332	244	122	212	5
INTERNAL STANDARDS					
1,4-Dichlorobenzene-d4	4.057	152	150	115	1
Naphthalene-d8	5.302	136	68		2
Acenaphthene-d10	7.556	164	162	160	3
Phenanthrene-d10	9.747	188	94	80	4
Chrysene-d12	13.887	240	236	120	5
Perylene-d12	15.858	264	265	260	6

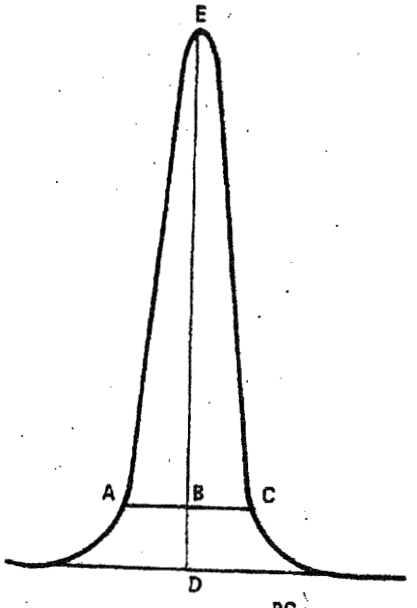
APPENDIX B- TARGET COMPOUNDS

APPENDIX IX TARGET LIST

PARAMETER	RT	Quant Ion	Secondary Ions			ISTD
1,4-Dioxane	1.933	88	58	45		1
Pyridine	2.178	79	52	51		1
2-Picoline	2.664	93	66			1
1,4-Benzoquinone	**4.466**	54	108	82		1
N-Nitrosomethylethylamine	2.739	88	42	43	56	1
Methyl methanesulfonate	2.964	80	79	65		1
N-Nitrosodiethylamine	3.268	102	42	44	57	1
Ethyl methanesulfonate	3.503	79	109	97	45	1
N-Nitrosopyrrolidine	4.481	100	41	42		2
Acetophenone	4.486	105	77	51		2
N-Nitrosomorpholine	4.497	56	86			1
O-Toluidine	4.529	106	107	79		2
Phorate	9.123	75	121			4
N-Nitrosopiperidine	4.796	114	42	55	56	2
O,O,O-Triethyphosphorothioate	5.122	65	97	93		2
2,6-Dichlorophenol	5.469	162	164	98		2
Hexachloropropene	5.507	213	211	215	117	2
1,2,4,5-Tetrachlorobenzene	6.447	216	214	179		3
a,a-Dimethylphenethylamine	5.619	58	91	42		2
N-Nitroso-di-n-butylamine	5.875	84	57	41		2
1,4-Phenylenediamine	5.864	108	80	107		2
Safrole	6.094	162	104	135	103	2
Isosafrole	6.757	162	104	131		2
1,1-Biphenyl	6.805	154	76			3
1,4-Naphthoquinone	7.056	158	104	76		3
m-Dinitrobenzene	7.317	168	76	50		3
Pentachlorobenzene	7.916	250	248	252	215	3
1-Naphthylamine	8.001	143	115	116		3
2-Naphthylamine	8.113	143	115	116		3
2,3,4,6-Tetrachlorophenol	8.140	232	230			3
5-Nitro-o-toluidine	8.466	152	77	106		3
Thionazin	8.471	107	96	97		4
Sulfotepp	9.032	97	65			4
1,3,5-Trinitrobenzene	9.091	213	74	120		4
1-Diallate	9.118	86	43	234		4
Phenacetin	9.161	108	109	179		3
2-Diallate	9.235	86	43	234		4
Dimethoate	9.396	87	93	125		4
4-Aminobiphenyl	9.556	169	168	170		4
Pronamide	9.748	173	175	145		4
Pentachloronitrobenzene	9.748	237	295	142	214	4
Disulfoton	9.935	88	60			4
Dinoseb	9.957	211	163	147		4
Methyl parathion	10.517	109	125			4
4-Nitroquinoline-1-oxide	11.094	174	101	128	75	4
Parathion	11.158	109	97			4
Famphur	**13.178**	218	93	125		4

Methapyrilene	11.388	97	58	191		4
Aramite-1	12.435	185	191	319		5
Aramite-2	12.563	185	191	319		5
p-Dimethylaminoazobenzene	12.638	120	225	77		5
Chlorbenzilate	12.745	139	251	75		4
Kepone	**17.739**	272	270	237		5
3,3'-Dimethylbenzidine	13.167	212	196	106		5
2-Acetylaminofluorene	13.562	181	180	223		5
7,12-Dimethylbenz(a)anthracene	15.438	256	239	241		6
Hexachlorophene	15.747	196	198			6
3-Methylcholanthrene	16.324	268	252	253		6
INTERNAL STANDARDS						
1,4-Dichlorobenzene-d4	4.102	152	150	115		1
Naphthalene-d8	5.346	136	68			2
Acenaphthene-d10	7.601	164	162	160		3
Phenanthrene-d10	9.802	188	94	189		4
Chrysene-d12	13.926	240	236	120		5
Perylene-d12	15.902	264	265	260		6

FIGURE 1 – Tailing Factor Calculation



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

ORGANOCHLORINE PESTICIDES AND PCBs BY GC

(Methods: EPA 608, 8081A, and 8082)

Technical Approval:

Andrea Sod — 03/22/04 (Date)

Title: Quality Assurance Manager
STL Savannah

Safety Approval:

Ernest B. Wood — 03/22/04 (Date)

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated pesticides and polychlorinated biphenyls (PCBs) as Aroclors in various matrices. Appendix A contains an example of the retention time order for the single peak pesticides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 608, 8081A, and 8082.

1.2 The routine target compounds, reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria are listed in the current revision of the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Environmental samples are prepared using matrix-specific procedures (see Section 9). The solvent is evaporated, the residue exchanged into hexane, and the sample adjusted to a final volume of 10mL or less. The preparation may also incorporate Florisil, copper (sulfur), acid (PCBs only), or gel permeation chromatography (GPC) cleanups. Analysis of the extract is routinely performed on a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. Quantitation may be performed using the external or internal standards calibration technique.

2.2 Method Clarifications / Default Procedures

General Clarification: The procedures for chlorinated pesticides (8081A) and PCBs (8082) are given as separate methods in Update III of SW-846. In previous updates, pesticides and PCBs were included in a single method; pesticides and PCBs are still included in the scope of EPA Method 608. The extraction and the analysis are combined in this SOP 1) to reduce the time of extraction and analysis; and 2) to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract can be subjected to the acid cleanup and reanalyzed. Note that if the list of target analytes includes only a limited list of components (i.e. Toxaphene, Chlordane, or PCBs), these procedures may be abbreviated to address only the analytes of interest.

Bracketing Sample Extracts: The laboratory's default procedure for continuing calibration verification stems from EPA Method 8000 and is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in an agency or client QAPP, in an STL pre-project plan, or in the method. EPA method 8081 specifically states to perform bracketing CCV every 12 hours (or 20 samples) and at the end of the analytical sequence; therefore, the requirement for capping CCV has been incorporated into this SOP.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Quantitation of QC Items: The default procedure for the analysis and evaluation of QC items (method blank, LCS, and MS/MSD) is to analyze these items on one of the instruments used to analyze the associated samples.
Dilutions: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the

upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.3 This method is based on the guidance in SW-846 Methods 8000B, 8081A, and 8082, and 40 CFR 136 Method 608.

2.4 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Acetone and hexane are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be thoroughly cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. See Section 9 for a table summary of the cleanups that may be employed to eliminate or reduce interferences. If matrix interferences continue after a cleanup has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank and laboratory control standard must also be subjected to the cleanup.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Aqueous samples are collected in 1-L glass containers with Teflon-lined caps. Soil/sediment samples are collected in wide mouth glass jars equipped with Teflon lined caps. No preservative is added. The samples are iced at the time of collection and maintained at 4°C (less than 6°C with no frozen samples) until extraction. Extraction must be performed within 7 days for aqueous samples and within 14 days of sampling for soils/solids. The extracts must be stored at 4°C (less than 6°C) and must be analyzed within 40 days of extraction.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with single or dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

CLP I fused silica capillary column 30 M x 0.53 mm ID x 1.5 µm film
CLP II fused silica capillary column 30 M x 0.53 mm ID x 0.83 µm film

6.4 Microsyringes, appropriate volumes

6.5 Volumetric flasks, Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps - compatible with the autosampler

7.0 REAGENTS

All reagents must be tracked in accordance with SOP AN44: *Reagent Traceability*.

Hexane - pesticide grade, for preparation of standards

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*. General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation*.

The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined single peak pesticides, technical chlordane, toxaphene, and the Aroclors.

If internal standard calibration is used, each calibration standard must contain the same concentration of the internal standard(s). The recommended concentration range for the internal standard(s) is 0.050 to 0.10µg/mL.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in the following SOPs:

PROCEDURE	MATRIX	STL SOP
Continuous Liquid-Liquid extraction	aqueous and leachates	EX30
Ultrasonic extraction	soils and sediments	EX40
Waste dilution	Waste samples (oils, products, etc)	EX42
Zymark extract concentration	All extracts	EX50

CLEAN-UP PROCEDURE	STL SOP	APPLICATION	EFFECTIVENESS
Florisil	EX62	Pest/PCBs	Eliminates polar non-target compounds
Sulfuric acid	EX60	PCBs	Eliminates some unsaturated hydrocarbon interferences
Copper	EX60	Pest/PCBs	Eliminates elemental sulfur
GPC	EX61	Pest/PCBs	Eliminates high molecular weight non-target compounds and sulfur

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions
The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of pesticides and PCBs. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Injector: 220 – 240°C
Detector: 300 – 320°C
Carrier Gas Flow: Helium at 5 mL/min (per column)
Make-up Gas Flow: Nitrogen at 25 mL/min (per detector)-see manufacturer's recommended flows

Example chromatogram temperature program:

Initial Temp:	160°C
Initial Hold:	4.0 min
Program Rate:	10°C/min
Final Temp:	270°C (hold for 10 minutes)
Injected Volume:	2-4µL - 1-2µL per column (single injection into guard column and "Y" splitter)

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize each analytical system.

10.2 Column Evaluation (608 and 8081A)

10.2.1 PEVAL Breakdown Standard

The column(s) must be evaluated prior to the analysis of the calibration standards and once every 12 hour clock. (NOTE: For EPA 608, this column evaluation is required once every 24 clock.) The column evaluation is performed by injecting a PEVAL standard that contains Endrin and p,p'-DDT and calculating the percent breakdown of these compounds. The standard used for determining the percent breakdown must not contain any compounds that coelute with Endrin, DDT, or any of the corresponding breakdown products.

NOTE: This column evaluation does not have to be performed if PCBs only are the target compounds. PCBs are stable and not subject to breakdown in the injection port.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Response(Endrin Aldehyde + Endrin Ketone)}}{\text{Response(Endrin + Endrin aldehyde + Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Response(DDE + DDD)}}{\text{Response(DDT + DDE + DDD)}} \otimes 100$$

The response (area or height) must be used to evaluate the breakdown. Do not use concentrations and do not "undetect" peaks that are below the RL or MDL. All peaks detected by *the data system must be included in the percent breakdown calculation.*

Breakdown Criterion
The breakdown for each compound must be less than 15%. If the breakdown exceeds 15%, the instrument will require column and/or injector port maintenance. The maintenance may include but is not limited to replacing the septum, clipping the front of the guard column, replacing the glass injector sleeve, and scrubbing (cleaning) the injector port.

10.2.2 If the instrument has not been in use for more than one day, a "priming" analysis may be beneficial. The analysis of a relatively high concentration pesticide or PCB standard may help to stabilize the response of the very sensitive EC detector. Inject a standard that is about 10x the concentration of the highest calibration standard and allow the instrument to cycle through the temperature program. It is not necessary to acquire the data but the baseline should be monitored before and after the priming analysis to gauge the condition of the detector. A hexane blank should be analyzed after the analysis of the priming standard and before the % breakdown check.

NOTE: The "priming" standard should be injected manually to avoid contaminating the autosampler syringe.

10.3 Initial Calibration

Initital calibration must be performed in accordance with SOP AN67: *Evaluation of Calibration Curves. Internal or external standard calibration techniques may be employed for the* determination of the concentration of pesticides and PCBs. Pentachloronitrobenzene (PCNB) or 2-Nitro-1-bromobenzene is recommended for use as internal standards; however, other compounds may be used.

Internal standard calibration should be used as the default. If matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

10.3.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for *establishing the analytical sequence is given in the SOP Summary.*

Note that the following offers two options for calibration and quantitation – average CF or regression curve. Only one needs be chosen per analyte.

10.3.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.3.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves*.

10.3.4 Initial Calibration Criteria:

600-series: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

8000-series: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The "grand mean exception" described below should be applied to 8081A and 8082 initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20%, with no individual compound exceeding 3X the ICAL criteria (60%).
NOTE: If a target compound that passes by the "grand mean exception" is detected the PM is notified via an anomaly report or case narrative.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If the regression *coefficient of the regression curve is greater than 0.99, the curve can be used to quantify*

samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order (quadratic) curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

A second source initial calibration verification standard must be performed in accordance with SOP AN67.

Calibration is verified at the frequency given in the SOP Summary. The following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported if appropriately qualified.

Analyze a mid-level standard. The concentration of the verification standard should be varied quarterly to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Verification Criteria

Response Criteria
If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

600-series: If the percent drift or percent difference is less than or equal to 15%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

8000-series: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compounds exceeds 3X (45%) the CCV criteria.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM *is notified via an anomaly report or case narrative.*

All samples must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no

target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (per SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses*.

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within –50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence
The analytical sequences for the 600- and 8000-series methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock.

For 8081 and 8082, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

For 608, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 24 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

If the internal standard calibration is used, the concentration(s) of the internal standard(s) must be the same in all calibration samples, field samples, and QC samples. A concentration of 0.050ug/mL to 0.10ug/mL (final extract concentration) is recommended.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to

provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks (such as phthalate esters, which elute in the same general retention time range as the pesticides and PCBs) or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses*. If internal standard calibration is used relative retention times, as described in Section 11.1.4, are used to identify the target compounds.

11.0 DATA ANALYSIS AND CALCULATIONS

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations*. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

11.1 Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be done when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) *is used to* determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed previously.

11.1.1 Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria
The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range ±50% of the response of the *internal standard in the associated CCV.*

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:
1) dilute the extract or perform sample extract cleanup to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria
A minimum of two surrogates is spiked into each sample and QC item prior to preparation. Decachlorobiphenyl (DCB) and 2,3,4,6-tetrachloro-m-xylene (TCMX) are the recommended surrogates. DCB should be evaluated as the primary surrogate; TCMX is evaluated if there is matrix interference with DCB.

NOTE: TCMX (or an alternate surrogate such as octachloronaphthalene) is to be used to evaluate data when AR1268 is detected. That is, TCMX or the alternate surrogate must pass the *surrogate recovery criterion in order to report the data without qualification.* AR1268 contains DCB and will bias the recovery when DCB is evaluated as a surrogate.

Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported with appropriate qualification. Refer to SOP AN02: *Analytical Batching and Evaluation of QC Data* regarding this issue.

Dilutions and Surrogate Recovery
The concentration range for surrogates is approximately 0.0025µg/mL to 0.080µg/mL. This should give up to a six-fold dilution to report surrogate recoveries, if the spiking level is 0.5µg/L and the lower limit of quantitation is 0.025µg/L. The lower recovery limit is 30%; therefore, the lowest acceptable concentration is 0.5*0.3=0.15µg/L. The highest dilution to report un-qualified results ("J" flag) would be 0.15µg/L/0.025µg/L = 6 or a six fold dilution.

11.1.2 Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the reporting limit standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected on the confirmation column or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

11.1.3 If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, the analyte is confirmed; however, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Report the result that is most reasonable for the sample. The lower result is reported as the default when the %RPD is >40. Flag the result to note the disparity (P flag) between the columns.

NOTE: The default guidance instructs the analyst to report the highest result when the %RPD <=40% and to report the lowest result when the %RPD exceeds 40%. The guidance uses RPD to determine if matrix interference is present. If the RPD is <=40%, the results between the two columns are essentially the same, and no matrix interference is deemed present. If the RPD exceeds 40%, a matrix interference is most likely present, and the lower result is reported as the default. This guidance does not prohibit reporting the higher result when the %RPD exceeds 40; however, this guidance is intended to preclude reporting clearly unreasonable data. It is this laboratory's experience that a majority of matrix interferences are positive and that reporting the lower of the two results is reasonable when the %RPD exceeds 40.

Alternatively, perform additional extract cleanup (sulfur, florisil, etc.) or if cleanup is not feasible or there is a low probability that cleanup will help, dilute the extract to a level that removes the interference and report the RL from this dilution.

The table below gives default guidance for evaluating hits.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recoveries meet the acceptance criteria and peaks fall within the middle of the retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P" No dilution required.
	<E	>E		

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report results less than MDL as <RL.

MS/MSD Evaluation
If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.5 Identification "Tools"
Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds in accordance with SOP SM06: *Guidelines for SIM Analysis by GC/MS*.

11.1.5.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.1.5.2Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

11.1.6 Qualitative Analysis of Multiple Peak Compounds

Identification of multi-peak pesticides (Toxaphene and Technical Chlordane) and PCBs as Aroclors is based on the recognition of their chromatographic patterns. Quantitation is performed using the area of characteristic peaks in the sample and standard using external or internal calibration procedures.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be warranted. Suggested cleanup options are given in Section 9.

11.1.6.1PCBs as Aroclors

PCBs are generally reported as Aroclors. The Aroclors have varying levels of PCB congeners with the last two numbers in the Aroclor designation indicating the weight percent of chlorine. For example, AR1221 is 21% chlorine by weight; AR1260 is 60% chlorine by weight. The 12- in the Aroclor designation represents the biphenyl molecule. The exception to this naming convention is AR1016, which is about 42% chlorine by weight. (Note that AR1016 and AR1242 have similar chromatograms - both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.)

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" of the Aroclor in the environment. The presence of multiple Aroclors will also complicate identification and quantitation. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper, and sulfuric acid, prior to analysis. STL SL SOP EX60 details this procedure.

NOTE: Do not use the acid cleanup on the entire extract if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer.

When a pattern matching an Aroclor is encountered, it may be quantitated using either the 3-5 characteristic peaks (recommended) or total area response. Total area quantitation should only be used as detailed below. Residues of either AR1016 or AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors. (i.e. if AR1660 was calibrated from 0.10µg/mL to 5.0µg/ml, and a sample extract was analyzed containing 10µg/ml of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 5.0µg/ml.) If a sample contains any of the single point Aroclors (that is, Aroclors other than AR1016 and AR1260), then that associated standard must be run within 72 hours of the sample to determine retention time shifts and pattern recognition.

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified or make a note in the case narrative if provided.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few peaks in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level.

NOTE: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

11.1.5.2 *Toxaphene*

Toxaphene is a mixture of chlorinated camphenes, which has a complex and characteristic pattern when analyzed by GC-ECD. A single Toxaphene standard is analyzed during the initial calibration for the purpose of pattern identification in samples. When a Toxaphene residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for Toxaphene should be analyzed. Alternatively, single points may be prepared with Toxaphene concentrations within 2x the Toxaphene quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the Toxaphene standard(s), the samples are re-analyzed using this standard(s) for quantitation. Note that when analysis of Toxaphene-containing samples occurs over an

extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

If the sample and standard chromatograms agree well, Toxaphene is quantitated using 5 characteristic peaks (similar to the PCB approach, above).

When Toxaphene is determined using the 5-peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionately larger or smaller in the sample compared to the standard.

In the 5-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. A minimum of three peaks must be used to determine the calibration factor. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 5 resulting concentrations are averaged to provide the final result for the sample.

11.1.5.3 Chlordane

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α and γ Chlordane, trans-Nonachlor, Heptachlor, and Heptachlor-epoxide. The α and γ Chlordane isomers are the most prevalent and their detection as single components is a good indicator that Technical Chlordane may be present.

When the GC pattern of the residue resembles that of the Technical Chlordane standard, quantitate Chlordane residues by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

When a Technical Chlordane residue is detected in sample(s), sample analysis is stopped. A calibration curve with at minimum of 5 points bracketing the instrument calibration range for technical chlordane should be analyzed. Alternatively, single points may be prepared with technical chlordane concentrations within 2x the Technical Chlordane quantity detected in the samples. Generally, the calibration curve option is simpler. After analysis of the technical chlordane standard(s), the samples are re-analyzed using these standard(s) for quantitation. Note that when analysis of technical chlordane-containing samples occurs over an extended time, the calibration factor should be verified or regenerated every 12 hours for method 8081, and every 24 hours for method 608.

NOTE: These procedures are not necessary if the lab is reporting chlordane as the α and γ chlordane isomers, not as the technical product.

11.2 Refer to SOP AN67: *Evaluation of Calibration Curves* for the calculations of sample concentrations.

12. 0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 Analytical Batching

QC data must be evaluated against the precision and accuracy criteria set forth in the Laboratory Quality Manual and SOP AN02: *Analytical Batching and Evaluation of QC Data*. These criteria are summarized in the SOP Summary included in Appendix C

SOP AN02 also provides guidance for establishing and evaluating QC items to be included in an analytical batch.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures*. CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability*.

13.2 Method Detection Limit

The method detection limit must be determined annually for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL)*.

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management*.

The following waste streams are produced when this method is carried out:

- Flammable waste (hexane and methanol from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Excess water samples. Dispose according to characterization on sample disposal sheets. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for storage.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Methylene chloride extracts. Dispose according to characterization on sample disposal sheets. If non-hazardous, transfer extract to chlorinated waste container. If hazardous, transfer to hazardous waste department for storage.

16.0 REFERENCES

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A: CHLORINATED PESTICIDES AND PCBs STANDARDS

Routine Targets

Target compound	STD ISMA-1 (ug/mL)	STD ISMA-2 (ug/mL)	STD ISMA-3 (ug/mL)	STD ISMA-4 (ug/mL)	STD ISMA-5 (ug/mL)
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040
g-BHC(Lindane), Heptachlor, Heptachlor epoxide, Endosulfan I, a-BHC, b-BHC, d-BHC, a-Chlordane, g-Chlordane	0.0050	0.010	0.020	0.030	0.050
Dieldrin, p,p'-DDT, Endosulfan II, Endrin aldehyde, Methoxychlor, p,p'-DDE, Endrin p,p'-DDD, Endosulfan sulfate, Endrin ketone	0.010	0.020	0.040	0.060	0.10

DDT/Endrin Breakdown Evaluation Standard

Pesticide Evaluation Standard	CONC (ug/mL)
Endrin,	0.040
P,P'-DDT	0.040

Appendix IX Targets

Target compound	APIX-1 (ug/mL)	APIX -2 (ug/mL)	APIX -3 (ug/mL)	APIX -4 (ug/mL)	APIX -5 (ug/mL)
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040
Isodrin	0.0050	0.010	0.020	0.030	0.050
Chlorobenzilate	0.050	0.10	0.20	0.50	1.0
Kepone	0.025	0.050	0.10	0.20	0.50

APPENDIX B

Technical Chlordane Five-point Curve

STOCK STANDARD	TCHLOR -1	TCHLOR -2	TCHLOR -3	TCHLOR -4	TCHLOR -5
Technical Chlordane	0.10	0.20	0.40	0.60	0.80
TCMX, DCB OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

Toxaphene Five-point Curve

STOCK STANDARD	TOX -1	TOX -2	TOX -3	TOX -4	TOX -5
Toxaphene	0.10	0.20	0.40	1.0	2.0
TCMX, DCB, OCN (surr)	0.0025	0.0050	0.010	0.020	0.040

PCBs as Aroclors

AR1660 Standards

Calibration Std	AR1016 (ug/mL)	AR1260 (ug/mL)	Surrogates (ug/mL)
AR1660-1	0.10	0.10	0.0025
AR1660-2	0.20	0.20	0.0050
AR1660-3	0.50	0.50	0.010
AR1660-4	1.0	1.0	0.020
AR1660-5	2.0	2.0	0.040

Single Point Aroclor Calibration Standards

Calibration Standard	Single Pont Concentration (ug/mL)	Surrogate Concentrations (ug/mL)
AR1221	1.0	0.020
AR1232	1.0	0.020
AR1242	1.0	0.020
AR1248	1.0	0.020
AR1254	1.0	0.020

If required, five point curves for AR1221, AR1232, AR1242, AR1248, and AR1254 are prepared at the same concentrations as the AR1660 curve.

Appendix B - METHOD SUMMARY

HOLD TIMES

MATRIX	Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Aqueous	None; 4C	1-L amber	7 days	40 days
Soil/ Sediment	None; 4C	500-mL	14 days	40 days
Waste	None; 4C	Glass	14 days	40 days
TCLP	None; 4C	1-L amber	7 days to leach / 7 days (after leaching procedure)	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION

Aqueous: Approximately 1L of sample (contents of container) using continuous at pH 5-9 with methylene chloride; exchange to hexane and concentrate to final volume of 10mL.
Soil/Solids: Approximately 30g of sample using sonication with 1:1 acetone/hexane or 1:1acetone/methylene chloride; Concentrate to final volume of 10mL in hexane
Wastes: Approximately 1g of sample diluted to final volume of 10mL with hexane

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:	Aqueous (ug/L)	Soils (ug/kg)
Tetrachloro-m-xylene	0.50	15
Decachlorobiphenyl	0.50	15
OCN	0.50	15

608 BATCH QC

Method blank
LCS/LCSD- full target list of single peak analytes @ 0.20ug/L
MS/MSD- full target list of single peak analytes @ 0.20ug/L

8081/8082 BATCH QC

Method blank
LCS - LQM subset
MS/MSD - LQM subset

Parameter	Aqueous (ug/L)	Soils (ug/kg)
Lindane	0.20	6.0
Aldrin	0.20	6.0
Heptachlor	0.20	6.0
Dieldrin	0.50	15
Endrin	0.50	15
p,p'-DDT	0.50	15

Appendix C

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock for 8081/8082 and a 24-hour clock for 608.

ANALYTICAL SEQUENCE – 608, 8081A, and 8082 (Pesticides and PCBs)

STANDARD / SAMPLES
Initial Calibration - 5 point single peak pesticides 5 point AR1660 (*Note 2) 1 point Toxaphene (*Note 1) 1 point Technical Chlordane (*Note 1) 1 point remaining Aroclors
Instrument Blank
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
ICV (Initial Calibration Verification) - 2nd source calibration verification
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank
Up to twenty sample extracts and QC and/or 12 hours for 8000-series Up to twenty sample extracts and QC and/or 24 hours for 600-series
PEVAL - Endrin/p,p'-DDT Breakdown Check (*Note 3)
CCV (Continuing Calibration Verification) - Midpoint single peak pesticides and midpoint AR1660 (*Note 2)
Instrument Blank

*Note 1 – If Toxaphene or Technical Chlordane is detected in a sample, a full 5-point ICAL must be performed and the sample reanalyzed.

*Note 2 - A mixture of AR1016 and AR1260 will be used to calibrate and verify the response for PCBs. Mid-level standards of the remaining Aroclors must be analyzed every 72 hours for pattern recognition and retention time.

*Note 3 – PEVAL Breakdown Check is not required for PCB-only analyses

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
p,p'-DDT and Endrin breakdown check -required for 8000-series, recommended for 600-series	Initially and every 12 hours	% breakdown of both compounds less than 15%	-re-analyze check solution -perform injector port and/or column *maintenance and re-analyze*
Initial Calibration- 600-series: 3 point minimum with lowest point at RL 8000-series: 5 point minimum with lowest point at RL	Initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	**600-series:** 1) RSD of each target <= 10%; OR 2) plot regression curve CC>=0.99 for each target **8000-series:** *1) RSD of each target* <= 20%; OR 2) plot regression curve CC>=0.99 for each target	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	After every twenty sample analyses and at the end of the sequence *8000-series: the capping* standard must be injected within 12-hours of the PEVAL after ICAL, within 12 hours of the previous CCV 600-series: the capping standard must be injected within 24-hours of the PEVAL after ICAL, within 24-hours of the previous CCV	-Percent difference or drift <= 15% (see SOP for use of "*grand mean*") -Response of the internal standard must be within a range of +/–50% of the mid-level standard (CCV) in the ICAL	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards

Appendix C

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Lab control sample (LCS)	*Per batch* (If MS/MSD cannot be performed, the LCS must be performed in duplicate)	*Recoveries within LQM limits*	*-Evaluate chromatogram and integrations.* Check calculations. -Reanalyze - Follow guidance in SOP AN02
Matrix spik e(MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02
Internal Standard Response	*All samples, method blanks, and QC*	-response within a factor of +/–50% of the previous CCV -retention time within window defined by previous CCV	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data
Surrogates	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze *- Follow guidance in SOP AN02*
Dilutions	When extract concentration exceeds calibration range	Report dilution where highest concentration target is in upper half of calibration	-Dilute sample to bring highest concentration into upper half of calibration range. Report all other targets from this dilution. -If lower RL required, prepare dilution 1/10 of dilution that puts highest concentration target into upper half of calibration range.

Appendix C

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Retention time window determination	Annually and with each new column. See guidance in SOP AN66	See guidance in SOP AN66	Use guidance in SOP AN66: *Determination of Retention Time Windows in Gas Chromatographic Analyses*
Demonstration of Capability	Initially and then annually per work group or analyst	Within the 600- or 8000-series method limits (see SOP CA92 and LQM)	-Reanalyze QC sample for the targets that failed to meet the criteria (see SOP CA92)
Method detection limit (MDL)	Annually See SOP CA90	Evaluate data using criteria SOP CA90	-Evaluate data. Check calculations. -Reanalyze MDL samples.

SEVERN TRENT STL

SOP SUMMARY FORM

SOP#: SG45:03.17.04:8	**SOP Description**: ORGANOCHLORINE PESTICIDES AND PCBs BY GC

Revisions:

___Minor _X_Significant ___Complete Re-write ___New SOP

Summary of Revision(s):

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information to be consistent with current STL Savannah format
- Updated Materials and Apparatus listing
- Added Quality Control, Method Performance and Waste Management information
- Removed Appendix A, *retention time table*
- Combined 8000-series and 600-series QC information into Appendix B
- Added cap to Grand Mean criteria per SOP AN67
- Added ICV requirement per SOP AN67
- Added requirement that alternate surrogate to DCB must be evaluated if AR1268 is detected
- Removed total area quantitation option for PCBs and Toxaphene, no longer performed
- Removed option NOT to perform capping CCV for internal standard analyses. Although method 8000 allows this option, Method 8081/8082 explicitly states that capping CCV must be performed.
- Removed reference to Separatory Funnel Extractions, no longer performed
- Removed reference to Permanganate clean-ups, no longer performed

IDOCs Required:	___Yes	_X_ No	__NA
MDLs Required:	___Yes	_X_ No	__NA

SOP Implementation Date: 04.17.04

Target Training Completion Date: 04.17.04

Approved by: Andrea Sal

Title: Quality Assurance Manager

Date: 03/22/04

Division Approval:

Title: Laboratory Director

Date: 3/22/04

ELEMENTS BY ICP (200.7 and 6010B)

Approved by:

R. Wayne Potts — 14 March 2001
Date

Title: Technical Manager, QA

STL X Savannah __ Tallahassee __ Mobile __ Tampa West

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP or TCLP), and waste samples after digestion.

1.2 Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element. The reporting limit (RL) for each element, the method detection limit (MDL) for each element, and the accuracy and precision criteria for each element are in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah, STL Tallahassee, STL Mobile, and STL Tampa West.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and exited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

2.2 Definitions

ICP -inductively coupled (argon) plasma; sometimes referred to a "ICAP"
TCLP-toxicity compound leaching procedure
EP (tox)-extraction procedure (toxicity)
Analytical Spike or Post-Digestion Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed
RL - reporting limit, the lowest calibration standard or the sample equivalent of the lowest calibration standard; published in LQM or project-specific quality assurance plan (QAPP); sometimes referred to as the "practical quantitation limit(PQL).
MDL - method detection limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM

2.3 This method is based on EPA Method 200.7 and SW-846 Method 6010B. Note that EPA has promulgated two versions of method 200.7-one for NPDES samples and one for drinking water. The calibration sequence for drinking water by 200.7 requires a multi-point curve with a minimum of three standards and a calibration blank.

3.0 SAFETY

3.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.2 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

3.3 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.

3.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.

4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions(MSA), or use of an internal standard

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Aqueous Samples

5.1.1 Liquid samples are routinely collected in 250-mL or 500-mL plastic containers. The sample is preserved with HNO_3 to a pH <2. The sample must be digested and analyzed within 6 months of collection. Samples may be stored at room temperature.

5.1.2 Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

5.2 Soil/Sediment Samples
Soil and sediment samples are routinely collected in 500-mL plastic containers. The sample is iced at the time of collection and is stored in the lab at 4C (less than 6C but not frozen) until time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

5.3 TCLP or EP Toxicity Leachate Samples
The leachate is transferred to a plastic container after the extraction procedure. The sample is preserved with HNO_3 to a pH <2. The leachate sample must be digested and analyzed within 6 months of completion of the leaching procedure.

5.4 Waste Samples
Waste samples are routinely collected in 500-mL plastic containers. The sample must be digested and analyzed within 6 months of collection.

6.0 APPARATUS AND MATERIALS

6.1 Thermo Jarrell Ash TJA ICAP61E-trace, or other suitable inductively coupled plasma emission spectrometer with data system

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Volumetric flasks

6.6 Pipettes

7.0 REAGENTS

Reagents are tracked in accordance with STL SOP AN44:*Reagent Traceability.*

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with STL SOP AN35.

7.2 Nitric acid (HNO_3)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.4 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. All preparation steps must be in accordance with STL SOP AN41: *Standard Materials Traceability.* SOP AN43 contains guidance for the preparation of standards.

8.1 Recommended concentrations for the calibration standards are given in Table 1. Appendix A contains examples for the preparation of the initial calibration and calibration verification standards for both 6010 and 200.7. if the laboratory uses "recipes' other than those listed in Appendix A, the recipe must be documented in the standard material traceability logbook or as controlled posting. All standards must have be prepared in 5% hydrochloric acid and 1% nitric acid by volume.

NOTE: Standards must be prepared every six months "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

8.2 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L)

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 5% by volume and the nitric acid concentration at 1% by volume.

9.0 SAMPLE PREPARATION

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SOP
Aqueous and leachate samples	ME50
Soils and Sediments	ME51
Wastes and oils	ME51

10.0 ANALYSIS PROCEDURE

The analytical sequence, including standardization and calibration verification, is included in the SOP Summary in Appendix A. The SOP Summary also included the acceptance criteria for QC, including recommended corrective actions.

10.1 Initial Calibration/Standardization

10.1.1 Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off, allow 2 hours warm up time.

10.1.2 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

10.1.3 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

10.1.4 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within +/- 5% of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

10.1.5 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

10.1.5.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the +/- 10 % of the true value.

10.1.5.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the +/-5.0 % of the true value. **When performing 200.7 work, note that this solution should be prepared fresh weekly.**

10.1.5.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the RL.

10.1.6 The RL/PQL Check Solution is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL). The determined concentration must within +/- 50% of the true concentration.

10.1.7 The ICP Interference Check Sample is analyzed. The concentrations of the target analytes must be within 20% of the true concentrations. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

10.2 Continuing Calibration Verification (CCV)

10.2.1 The calibration of the ICP must be verified every 10 samples by the analysis of the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

10.2.1.2 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/- 10 % of the true value.

10.2.1.2 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within the +/-5.0 % of the true value.

10.2.1.3 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

10.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

10.3 Sample Analysis

10.3.1 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.2 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/CALIBRATION OF THE ICP)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, ICSA, ICSAB, CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.3 Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.3.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

10.3.3.2 The amount of sample digestate needed to prepare the desired dilution is determined form the following equation:

$$V_{digest} = \frac{Vf_{vol}}{DF}$$

where
Vf_{vol} = final volume of diluted sample (mL)
V_{digest} = volume of sample digestate used to make the dilution (mL)

10.3.3.3 The dilution factor is calculated as follows:

$$DF = \frac{Vf_{vol}}{V_{digest}}$$

where
Vf_{vol} = final volume of diluted sample extract (mL)
V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE
A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

10.4 Dilution QC Check
A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.4.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.

10.4.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.4.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

10.4.4 If the results of the dilution are within ± 10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than ± 10%, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 10.4).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

10.5 Post-digestion Spike QC Check

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in 10.3, above.

10.5.1 Transfer 10mL of a digestate to a suitable vial.

10.5.2 Spike the sample with 0.10mL of ICP Matrix Spike I and 0.10mL of ICP Matrix Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.5.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

10.5.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

where

Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = theoretical concentration of spike (ug/L)
(See 10.2.5.2)

10.5.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA 4) Qualify all associated samples
<50% recovery	Dilute digestate and repeat spike. Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples) If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

10.5.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences.

10.6 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a solution of known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = Volume of spike solution

Example:		
	Sample concentration (S_2):	523 ug/L.
	Spike solution concentration (C_s):	50,000 ug/L
	Volume of spike solution (V_s):	0.10mL
	Volume of sample aliquots (V_x):	10mL
	Spiked sample concentration (S_1):	951 ug/L

C_x = [(523)*(0.10)*(50,000)]/[(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

10.7 Determination of Linear Range of the ICP

The linear range must be determined a minimum of once per year. Divisions performing CLP analyses are required to determine and document the linear range quarterly. Documentation of the linear range study must be kept on hand and be available for inspection. A summary of the linear range study must be available to the bench analyst.

10.7.1 Profile and calibrate the ICP as described in Section 10.1

10.7.2 Prepare individual standards at concentrations that are expected to define the linear range of the instrument. Use the concentrations in Table 1 for guidance. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

10.7.3 Analyze the standards following the analytical sequence described in Section 10.3. Verify the calibration after every 10 analyses.

10.7.4 Compare the concentration of the linear range standard with its true concentration.

$$PercentDifference = \left| \frac{Ccal - Ctrue}{Ctrue} \right| \otimes 100$$

where
Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

If the percent difference is less than or equal to 5%, the linear range is confirmed at that concentration. If the percent difference is greater than 5%, repeat the analysis with a lower concentration.

The linear range may be extended by analyzing higher standards and evaluating the results against the 5% difference criterion. The linear range of the ICP for an analyte is the highest standard of that analyte that meets this criterion.

11.0 DATA ANALYSIS/CALCULATIONS

11.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$Concentration\,(mg/L) = ug/L(from\ printout) \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)-usually 50mL (0.050L)
V = volume of sample digested (L)
DF = dilution factor

11.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$Concentration(mg/L) = RLqap \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
RLqap = reporting limit from STL LQM (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The LQM Reporting Limits assumes:
F = 50mL, V = 50mL, and DF = 1

11.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = ug/L(from\ printout) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$$

where
F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
(for example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.)

11.2.2 The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$Concentration(mg/kg,dw) = RL_{qap} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L}\ x\ DF$$

where
RL(qap) = reporting limit from LQM
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

The LQM Reporting Limits assumes F = 0.100L (100mL), DF = 1, W = 0.0010kg (1.0g), and solids = 1.0

12.0 QUALITY ASSURANCE /QUALITY CONTROL

12.1 STL SOP AN02: *Analytical Batching* and the SOP Summary provide guidance on evaluating QC and sample data, including recommended corrective actions.

12.2 The method detection limit (MDL) is determined annually in accordance with STL SOP CA90. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP.

12.3 Determination of the Instrument Detection Limit (IDL)

The difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Section 8 of this SOP. See SOP CA91 for the procedures for the determination of the IDL.

12.4 The linear range of the ICP must be determined at least annually. The procedure for the determination is given in Section 10.7 of this SOP. If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard.

12.5 Interelement correction factors (IEC) for all elements must be determined annually. Use the manufacturer's guidance for determination of the IECs. The IECs must be verified at the beginning and end of each analytical sequence.

13.0 TROUBLESHOOTING AND PREVENTIVE MAINTENANCE

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
ICAP								
Pump Tubing	X							Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.

D = daily W = Weekly M = monthly Q = Quarterly SA = semi-annually A = annually AN = as needed

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Excess samples, reagents, and digests must be disposed of in accordance with SOP CA70:*Waste Management*.

15.0 REFERENCES

15.1 *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.

15.2 *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986.

15.3 *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	10	1.0/5.0	0.20	800	2.0	200
Antimony (Sb)	206.838	10	1.0/0.50	0.020	10	0.50	50
Arsenic (As)	189.042 193.696	1.0	1.0/0.50	0.010	25	2.0	200
Barium (Ba)	493.409	10	1.0/5.0	0.010	10	2.0	200
Beryllium (Be)	313.042	1.0	1.0/0.50	0.0040	10	0.050	5.0
Boron (B)	249.678	10	1.0/5.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	1.0	1.0/5.0	0.0050	10	0.050	5.0
Calcium (Ca)	317.933 315.887	10	1.0/5.0	0.50	800	5.0	500
Chromium (Cr)	267.716	10	1.0/5.0	0.010	25	0.20	20
Cobalt (Co)	228.616	1.0	1.0/0.50	0.010	25	0.50	50
Copper (Cu)	324.754	10	1.0/5.0	0.020	50	0.25	25
Iron (Fe)	259.940 271.441	10	1.0/5.0	0.050	800	1.0	100
Lead (Pb)	220.353	1.0	1.0/0.50	0.0050	5	0.50	50
Magnesium (Mg)	279.079	10	1.0/5.0	0.50	1000	5.0	500

TABLE 1							
Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Manganese (Mn)	257.610	10	1.0/5.0	0.010	50	0.50	50
Molybdenum (Mo)	202.030	1.0	1.0/0.50	0.010	50	0.50	50
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040	10	0.50	50
Potassium (K)	766.491	20	10/5.0	1.0	50	5.0	500
Selenium (Se)	196.026	10	1.0/5.0	0.010	25	2.0	200
Silver (Ag)	328.068	1.0	1.0/5.0	0.010	5.0	0.050	5.0
Sodium (Na)	588.995 330.231	10	1.0/5.0	0.50	20	5.0	500
Strontium (Sr)	421.552	10	1.0/5.0	0.010	100	0.50	50
Thallium (Tl)	189.042 190.801 377.572	10	1.0/5.0	0.010	30	2.0	200
Tin (Sn)	189.989	10	1.0/5.0	0.050	50	1.0	100
Titanium (W)	334.941	10	1.0/5.0	0.010	10	1.0	100
Vanadium (V)	292.402	10	1.0/5.0	0.010	50	0.50	50
Zinc (Zn)	213.856 206.200+	5.0	1.0/2.5	0.020	20	0.50	50

*For guidance only-instrument sensitivity will vary.

APPENDIX A
SOP SUMMARY

METHOD SUMMARY - ICP ANALYSIS

HOLD/STORAGE

Container	Minimum 250mL plastic bottle with plastic or Teflon-lined lid
Preservative	HNO3 to pH <2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples must be analyzed within six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Profile Instrument	Follow manufacturer's guidelines.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing highest concentration standard for each element.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the RL by analyzing a solution at the SL RL
Serial Dilution	Perform serial dilution (1/5) on a representative sample from each batch..
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ±10% 200.7 = within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value, 200.7-NPDES - within ± 5% 200.7-Drinking Water – within ±10%	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less that the RL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning and end of an analysis run	Determined values must be within ± 20% of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: STL LQM 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	|result| <RL or result <5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	|result| <RL or result <10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD - SW846	One set per batch of twenty samples or less	STL LQM	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	See section 10.3.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 10.4.5	
Detection Limit Check Solution	At the beginning and end of an analysis run	Recovery +/-50% of the true concentration.	Stop the analysis, fix the problem and reanalyze the affected samples.

APPENDIX B
EXAMPLES OF STANDARD PREPARATION

GENERAL INSTRUCTIONS

All calibration standards must contain 5% hydrochloric acid and 1% nitric acid by volume. The following table lists the volume of each acid needed to prepare the desired final volume of standard.

Final Volume of Standard (mL)	Volume of Hydrochloric acid (mL)	Volume of Nitric Acid (mL)
100	5.0	1.0
200	10	2.0
500	25	5.0
1000	50	10

For example, to prepare 500mL of a standard:

- Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask.
- Add 5.0mL of concentrated nitric acid (HNO_3) and 25mL of hydrochloric acid (HCl) to the volumetric flask.
- Add the volumes of the stock standards given in the table to the volumetric flask:
- Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature.

SINGLE POINT CALIBRATION STANDARDS FOR 6010

Calibration Standard 1-Calibration Blank (ICB, CCB)

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid (HNO_3) and 50mL of hydrochloric acid (HCl) to the volumetric flask. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature. Other volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

Calibration STANDARD 2

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	0.50	500	1.0
Arsenic(As)	1000	0.50		1.0
Molybdenum(Mo)	1000	0.50		1.0
Lead(Pb)	1000	0.50		1.0
Selenium(Se)	1000	5.0		10
Antimony(Sb)	1000	0.50		1.0
Thallium(Tl)	1000	5.0		10

Calibration STANDARD 3

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	0.50	500	1.0
Barium(Ba)	1000	5.0		10
Cadmium(Cd)	1000	0.50		1.0
Cobalt(Co)	1000	0.50		1.0
Chromium(Cr)	1000	5.0		10
Copper(Cu)	1000	5.0		10
Manganese(Mn)	1000	5.0		10
Nickel(Ni)	1000	2.5		5.0
Zinc(Zn)	1000	2.5		5.0

Calibration STANDARD 4

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	0.50	500	10
Iron(Fe)	10000	0.50		10
Boron(B)	1000	5.0		10
Strontium(Sr)	1000	5.0		10
Titanium (Ti)	1000	5.0		10

Calibration STANDARD 5

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Calcium(Ca)	10000	0.50	500	10
Potassium(K)	10000	1.0		20
Magnesium(Mg)	10000	0.50		10
Sodium(Na)	10000	0.50		10
Tin(Sn)	1000	5.0		10
Vanadium(V)	1000	5.0		10

MULTI-POINT INSTRUMENT CALIBRATION-200.7

For all drinking water samples (EPA 200.7) the ICP must be calibrated with a minimum of three standards and a blank. The following standards may be used for this purpose. With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the multi-point calibration of the ICP instruments.

High Standard.

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum (Al)	10000	1.0	1000	10
Antimony (Sb)	1000	1.0		1.0
Arsenic (As)	1000	1.0		1.0
Boron (B)	1000	10		10
Barium (Ba)	1000	10		10
Beryllium (Be)	1000	1.0		1.0
Cadmium (Cd)	1000	1.0		1.0
Calcium (Ca)	10000	1.0		10
Cobalt (Co)	1000	1.0		1.0
Chromium (Cr)	1000	10		10
Copper (Cu)	1000	10		10
Iron (Fe)	10000	1.0		10
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	1.0		10
Manganese (Mn)	1000	10		10
Molybdenum Mo)	1000	1.0		1.0
Nickel (Ni)	1000	5.0		5.0
Potassium (K)	10000	1.0		10
Selenium (Se)	1000	10		10
Silver (Ag)	1000	1.0		1.0
Sodium (Na)	10000	1.0		10
Strontium (Sr)	1000	10		10
Thallium (Tl)	1000	10		10
Tin (Sn)	1000	10		10
Titanium (Ti)	1000	10		10
Vanadium (V)	1000	10		10
Zinc (Zn)	1000	5.0		5.0

Mid-Level Standard- Prepare as the CCV is prepared.

Low-Level Standard- Prepare as the RL/PQL Check Standard.

Initial Calibration Verification (ICV) Solution

Element/Stock	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
SPEX QC19	(1)	5.0	500	(2)
SPEX QC 7	(1)	5.0		(2)
Tin(Sn)	1000	0.50		1.0
Strontium (Sr)	1000	0.50		1.0
Potassium (K)	10000	0.50		10(3)
Sodium (Na)	10000	0.45		10(3)

(1) SPEX QC19 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.
(2) The final concentrations of the various elements are the same as listed in Table 1. The SPEC QC solutions are diluted by a factor of 100 from the concentration listed on the certificate of analysis.
(3) These concentrations include the contribution from SPEX Solutions QC-7

Continuing Calibration Verification (CCV) Standard

(also used as midpoint of multi-point calibration for EPA 200.7)

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	10000	0.50	1000	5.0
Antimony (Sb)	1000	0.50		0.50
Arsenic (As)	1000	0.50		050
Boron (B)	1000	5.0		5.0
Barium (Ba)	1000	5.0		5.0
Beryllium (Be)	1000	0.50		0.50
Cadmium (Cd)	1000	0.50		0.50
Calcium (Ca)	10000	0.50		5.0
Cobalt (Co)	1000	0.50		0.50
Chromium (Cr)	1000	5.0		5.0
Copper (Cu)	1000	5.0		5.0
Iron (Fe)	10000	0.50		5.0
Lead (Pb)	1000	0.50		0.50
Magnesium (Mg)	10000	0.50		5.0
Manganese (Mn)	1000	5.0		5.0
Molybdenum (Mo)	1000	0.50		0.50
Nickel (Ni)	1000	2.5		2.5
Potassium (K)	10000	0.50		5.0
Selenium (Se)	1000	5.0		5.0
Silver (Ag)	1000	0.50		0.50
Sodium (Na)	10000	0.50		5.0
Strontium (Sr)	1000	5.0		5.0
Thallium (Tl)	1000	5.0		5.0
Tin (Sn)	1000	5.0		5.0
Titanium (Ti)	1000	5.0		5.0
Vanadium (V)	1000	5.0		5.0
Zinc (Zn)	1000	2.5		2.5

Reporting Limit (RL) Check Standard

(also used as low point in multi-point calibrations; e.g. EPA 200.7)

Preparation of RL/PQL Stock A-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Silver (Ag)	1000	0.10	100	1.0
Arsenic (As)	1000	0.10		1.0
Cadmium (Cd)	1000	0.050		0.50
Copper (Cu)	1000	0.20		2.0
Nickel (Ni)	1000	0.40		4.0
Lead (Pb)	1000	0.050		0.50
Selenium (Se)	1000	0.10		1.0
Thallium (Tl)	1000	0.10		1.0

Preparation of RL/PQL Stock B-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. Of Stock Std (mg/L)
Aluminum (Al)	10000	0.20	100	20
Boron (B)	1000	0.50		5.0
Barium (Ba)	1000	0.10		1.0
Beryllium (Be)	1000	0.040		0.40
Calcium (Ca)	10000	0.50		50
Cobalt (Co)	1000	0.10		1.0
Chromium (Cr)	1000	0.10		1.0
Iron (Fe)	10000	0.050		5.0
Magnesium (Mg)	10000	0.50		50
Manganese (Mn)	1000	0.10		1.0
Molybdenum Mo)	1000	0.10		1.0
Sodium (Na)	10000	0.50		50
Antimony (Sb)	1000	0.20		2.0
Strontium (Sr)	1000	0.10		1.0
Tin (Sn)	1000	0.50		5.0
Titanium (Ti)	1000	0.10		1.0
Vanadium (V)	1000	0.10		1.0
Zinc (Zn)	1000	0.20		2.0

Preparation of RL/PQL Stock C-ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume of Cal Std	Conc. Of Stock Std
Potassium(K)	10000	1.0	100	100

Preparation of the RL/PQL Check Solution-ICP

RL/PQL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A-ICP	5.0	500
Stock B-ICP	5.0	
Stock C-ICP	5.0	

ICP Interference Check Solutions

Preparation of ICP Interference Check Solution A

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200

Preparation of ICP Interference Check Solution AB

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25		500
Magnesium (Mg)	10000	25		500
Iron (Fe)	10000	10		200
Silver (Ag)	1000	0.10		0.20
Arsenic (As)	1000	0.050		0.10
Barium (Ba)	1000	0.25		0.50
Beryllium (Be)	1000	0.25		0.50
Cadmium (Cd)	1000	0.50		1.0
Cobalt (Co)	1000	0.25		0.50
Chromium (Cr)	1000	0.25		0.50
Copper (Cu)	1000	0.25		0.50
Manganese (Mn)	1000	0.25		0.50
Nickel (Ni)	1000	0.50		1.0
Lead (Pb)	1000	0.025		0.050
Antimony (Sb)	1000	0.30		0.60
Selenium (Se)	1000	0.025		0.050
Thallium (Tl)	1000	0.050		0.10
Vanadium (V)	1000	0.25		0.50
Zinc (Zn)	1000	0.50		1.0
Molybdenum (Mo)	1000	0.50		1.0
Tin (Sn)	1000	0.50		1.0

ICP Matrix Spiking Solutions

ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The certificate of analysis will list the concentrations of the analytes. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

Preparation of the ICP Matrix Spiking Solution 2

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100
Titanium (Ti)	1000	10		100

IDL/MDL Solution

The IDL/MDL solution is used in this procedure for two purposes:

1) To determine the Instrument Detection Limit (IDL) of each target compound on a quarterly basis (SOP CA91); and
2) To determine the Method Detection Limit (MDL) of each target compound on an annual basis (SOP CA90). MDLs should be digested straight and at 1:2 dilutions.

Preparation of IDL/MDL Stock A

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Silver (Ag)	1000	0.040	100	0.40
Arsenic (As)	1000	0.20		2.0
Barium (Ba)	1000	0.020		0.20
Beryllium (Be)	100	0.050		0.050
Cadmium (Cd)	1000	0.040		0.40
Lead (Pb)	1000	0.10		1.0
Antimony (Sb)	1000	0.20		2.0
Selenium (Se)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0

Preparation of IDL/MDL Stock B

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. Of Std. (mg/L)
Cobalt (Co)	1000	0.030	100	0.30
Chromium (Cr)	1000	0.10		1.0
Copper (Cu)	1000	0.10		1.0
Manganese (Mn)	1000	0.020		0.20
Molybdenum (Mo)	1000	0.040		0.40
Nickel (Ni)	1000	0.10		1.0
Tin (Sn)	1000	0.20		2.0
Vanadium (V)	1000	0.060		0.60
Zinc (Zn)	1000	0.10		1.0

Preparation of IDL/MDL Stock C

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Aluminum (Al)	10000	0.10	100	10
Calcium (Ca)	10000	0.10		10
Iron (Fe)	10000	0.10		10
Magnesium (Mg)	10000	0.10		10
Potassium (K)	10000	0.20		20
Sodium (Na)	10000	0.040		4.0

IDL/MDL Solution

Preparation of IDL/MDL Stock D

Element	Conc. Of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Boron(B)	1000	0.40	100	4.0
Strontium (Sr)	1000	0.040		0.40
Titanium (Ti)	1000	0.050		0.50
Sodium (Na)	10000	2.0		200

Preparation of the IDL/MDL Check Solution

IDL/MDL Stock	mL of RL/PQL Stock	Final Volume(mL)
Stock A	5.0	1000
Stock B	5.0	
Stock C	5.0	
Stock D	5.0	

The IDL/MDL Check Solution contains the following elements at the given concentrations:

Element	Concentration(mg/L)
Be	0.00025
Ba, Mn	0.0010
Co	0.0015
Ag, Cd, Mo, Sr	0.0020
Ti	0.0025
V	0.0030
Cr, Cu, Ni, Pb, Zn	0.0050
As, Sb, Se, Sn, Tl	0.010
Na(1), B	0.020
Ca, Fe, Mg	0.050
Al, K	0.10
Na	1.0

(1) If the wavelength for sodium is 588.995, the lower concentration (0.020mg/L) is used for the IDL./MDL check solution. In this case, only stocks A,B, and C are used to make the IDL/MDL Check Solution. IDL/MDL Check Solutions for B, Sr, and Ti are prepared and evaluated separately.

MERCURY PREPARATION AND ANALYSIS

(Methods: EPA 7470A, 7471A, and 245.1)

Technical Approval:

Andrea Seal — 01/30/04 (Date)

Title: QA Manager
STL Savannah

Safety Approval:

Ernest R. Watts — 02/03/04 (Date)

Title: EHSC
STL Savannah

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure to determine the concentration of mercury by cold vapor atomic absorption spectrophotometry (CVAA). This method contains the manual preparation and the analytical procedures for determination of mercury in aqueous liquids (water), surface and groundwaters, soils, sediments, sludges, wastes and leachates (EP or TCLP) after digestion.

1.2 The reporting limit (RL), method detection limit (MDL), and the accuracy and precision criteria are listed in the current revision of the *Laboratory Quality Manual* (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF THE METHOD AND DEFINTIONS

2.1 This method is based on the absorption of characteristic radiation at 253.7nm by mercury vapor. After digestion, to convert all forms of mercury to the same oxidation state, the mercury ions are reduced to mercury by the addition of stannous chloride and aerated from solution after passing through a mixing coil. The mixture passes through a gas/liquid separator and through a drying tube. The vapor is passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is measured as a function of absorbance

2.2 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

CVAA - cold vapor atomic absorption
TCLP – toxicity characteristic leaching procedure
EP Tox - extraction procedure toxicity
Analytical Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed; also called a post digestion spike
RL - reporting limit, the lowest calibration standard or the sample equivalent of the lowest calibration standard; published in LQM or project-specific quality assurance plan (QAPP); sometimes referred to as the "practical quantitation limit" (PQL).
MDL - method detection limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM

2.3 This method is based on the guidance provided in SW-846 methods 7000A, 7470A, 7471A, and EPA method 245.1 (Drinking Water version).

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, the Waste Management SOP, and this document.

3.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified

The making of aqua regia can produce toxic fumes and heat This procedure must be performed under a working fume hood.

The exhaust of the mercury analyzer must be vented or trapped so that mercury vapors do not enter the laboratory.

3 2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2ppm-TWA 4ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2 Chlorine is known to interfere with this analysis. Addition of extra potassium permanganate may be needed during the digestion of samples containing chloride. Also, the samples are not capped tightly during digestion so that excess chlorine can escape.

4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain mercury. Reagent blanks (method blanks) must be analyzed as a check on contamination due to sample digestion.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

5 1 Aqueous samples and TCLP/EP-TOX Leachate

5.1.1 Liquid samples are collected in 250-mL plastic or glass containers. The samples are preserved with HNO3 to a pH<2. Samples must be digested and analyzed within 28 days of collection.

5 1.2 Samples for dissolved mercury should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered The sample is stored at 4°C (less than 6°C, but not frozen) until filtration and preservation.

5.2 Soil/Sediment/Waste Samples

5 2.1 Soil and sediment samples are collected in 250-mL or 500-mL plastic or glass containers The samples are iced at the time of collection and stored at 4°C (less than 6°C but not frozen) until

the time of digestion and analysis. Samples must be digested and analyzed within 28 days of collection.

6.0 APPARATUS AND MATERIALS

6.1 Leeman Hydra AA or other suitable automated mercury analyzer with data system and printer

6.2 Nitrogen or argon gas supply and appropriate fittings

6.3 Pump tubing of appropriate sizes for use on the Hydra AA

6.4 Volumetric glassware for making standards and reagents

6.5 Test tubes of the two sizes to fit the Hydra AA autosampler

6.6 Water bath or heating block capable of maintaining a temperature of 95 ± 5°C

6.7 Digestion glassware

7.0 REAGENTS

All reagents must be tracked in accordance with SOP AN44: *Reagent Traceability*.

7.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity is monitored in accordance with SOP AN35: *Conductivity Checks for Laboratory Deionized Water*.

7.2 Nitric Acid (HNO_3), concentrated-reagent grade

7.3 Hydrochloric Acid (HCl), concentrated-reagent grade

7.4 Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.5 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 50g of $KMnO_4$ in 1000mL of DI water.

7.6 Sodium chloride-hydoxylamine sulfate solution: Dissolve 120g NaCl and 120g hydroxylamine sulfate in DI water in a 1-L volumetric flask and dilute to volume.

7.7 Potassium persulfate, 5% solution (w/v): Dissolve 50g potassium persulfate in 1000mL DI water.

7.8 Rinse Water, 5% HCl-1%HNO3 - to a clean 2-L bottle add 1-L of reagent water. Carefully add 100mL of concentrated hydrochloric acid. Carefully add 20mL of concentrated nitric acid. Dilute to a final volume of 2L. Other volumes may be utilized providing the reagent proportions remain the same.

7.9 Stannous chloride ($SnCl_2.2H_2O$) - reagent grade, suitable for mercury determination

7.10 Stannous chloride ($SnCl_2.2H_2O$) solution - to a clean 2-L volumetric flask add 100g of stannous chloride. Add approximately 400mL of reagent water Carefully add 500mL of concentrated hydrochloric acid. Add a stirring bar and stir on a stir plate until the stannous chloride is dissolved Remove the stirring bar and dilute to volume with reagent water

7.11 Magnesium perchlorate ($Mg(ClO_4)_2$) - used as a drying agent in the drying tube. The magnesium perchlorate should be as coarse as possible.

7.12 Sulfuric Acid (H_2SO_4), concentrated reagent grade

8.0 STANDARDS

All standards must be tracked in accordance with SOP AN41: *Standard Material Traceability*.

8.1 Commercial stock standard, 1000mg/L.

8.2 Independent stock standard, 1000mg/L.

8.3 Calibration standards

8.3.1 Mercury calibration intermediate stock standard, 10mg/L: Add 1mL of the commercial stock standard, 1000mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.3.2 Mercury intermediate working standard, 500µg/L: Add 5mL of the 10mg/L intermediate stock standard and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.3.3 Mercury Calibration Standards: Transfer 0.0, 0.02, 0.04, 0.1, 0.3, and 0.5mL portions of the intermediate working standards to a series of 125mL glass bottles. Add DI water from a graduated cylinder to each bottle to make a final volume of 50mL. This results in working standard concentrations of 0.0, 0.2, 0.4, 1.0, 3.0, and 5.0µg/L mercury. Mix well and add 2.5mL of concentrated H_2SO_4, 1.25mL of concentrated HNO_3, and 7.5mL of $KMnO_4$ solution and let stand at least 15min. Add 4mL of potassium persulfate and heat for 2 hours in a water bath at 95°C. Cool and add 3mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. The standards are ready for analysis. Larger volumes of standards may be digested as needed as long as reagent ratios are kept the same.

8.4 Calibration verification standards - Initial (ICV) and Continuing (CCV):

8.4.1 Verification intermediate stock, 5.0mg/L: Add 0.5mL of the Independent stock standard, 1000mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.4.2 Verification Working Stock, 250µg/L: Add 5mL of the verification intermediate stock, 5.0mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.4.3 Initial Calibration Verification (ICV) Standard, 3.0µg/L: Add 0.60mL of the verification working stock, 250µg/L, to a 125-mL glass bottle and add enough reagent water from a graduated cylinder to make a final volume of 50mL. The ICV is now ready to be digested. Other final volumes may be used as long as the reagent ratios are kept the same.

8.4.4 Continuing Calibration Verification (CCV) Standard, 2.5µg/L: Add 0.50mL of the verification working stock, 250µg/L, to a 125-mL glass bottle and add enough reagent water from a

graduated cylinder to make a final volume of 50mL. The CCV is now ready to be digested. Other final volumes may be used as long as the reagent ratios are kept the same

8.5 QC Standards

8.5.1 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Working Stock, 100µg/L: Add 2mL of the verification intermediate stock, 5.0mg/L, and 2.5mL of nitric acid to about 50mL of DI water in a 100-mL volumetric flask and dilute to volume.

8.5.2 MS/MSD, 1.0µg/L (0.05 mg/kg): To two portions of a selected sample (50mL for aqueous or 1.0-1.2g of a solid sample) add 0.5mL of the MS/MSD working standard. The MS/MSD is now ready for digestion. Where sufficient sample is available, at least 1 set of MS/MSDs should be prepared and analyzed with each batch of 20 samples or less.

8.5.3 Analytical Spike Stock Standard, 73.26µg/L: Add 2mL of the verification intermediate stock, 5.0 mg/L, and 2.5mL of nitric acid to about 50mL of DI water and dilute to 136.5mL final volume

8.5.4 Analytical Spike, 1µg/L: To a 10mL portion of digested sample, add 0.1mL of the Analytical Spike Stock Standard. One analytical spike is required per batch of twenty samples or less.

8.5.5 Lab Control Standard for Water Samples (LCSW) - 2.5µg/L: Add 0.50mL of the verification working stock, 250µg/L, to a 125mL glass bottle and add enough reagent water from a graduated cylinder to make a final volume of 50mL. The LCSW must be digested/prepared in the same manner as the samples. At least 1 LCSW must be digested with each batch of 20 samples or less.

8.5.6 Lab Control Standard for Soils Samples (LCSS): Weigh an appropriate weight of a certified solid reference standard into the digestion bottle, an example would be 0.10g of the NIST 2709 San Joquin Soil The lab control standard must be digested/prepared in the same manner as the samples.

9.0 SAMPLE PREPARATION

This section describes the manual digestion procedures for aqueous, soils/wastes, and biological matrices.

9.1 Liquid samples

9.1.1 Mix the sample thoroughly and add 50mL of sample or an aliquot of sample diluted to 50mL to a 125-mL glass bottle.

9.1.2 Add 1.25mL HNO_3, 2.5mL H_2SO_4, and 7.5mL of $KMnO_4$ solution to each sample. Shake well after each addition. Be sure the purple color of $KMnO_4$ persists for at least 15min If not, add up to three times more $KMnO_4$ solution. Equal quantities of $KMNO_4$ must be added to the LCS and MB.

9.1.3 Add 4mL of potassium persulfate to each sample, shake well, and place the samples in a water bath or block digestion apparatus at 95 ± 5°C for 2 hours.

9.1.4 Remove and allow the samples to cool. Add 3mL of hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$ Follow the analysis procedure below (Section 10)

9.1.5 If a different final volume is obtained (due to additional $KMNO_4$ or other reason) a dilution factor must be obtained in order to correct the final result.

9.2 Soil/Solid Samples

9.2.1 Homogenize the sample thoroughly and weigh between 1.0g and 1.2g wet weight of sample into a 125-mL glass bottle

9.2.2 Add 2.5mL DI water and 2.5mL aqua regia. Heat for 2min in water bath or digestion block at 95°C.

9.2.3 Allow the samples to cool to room temperature and add 25mL DI water and 7.5mL $KMnO_4$ solution to sample. Mix and heat for 30 minutes at 95 ± 5°C.

9.2.4 Allow the samples to cool to room temperature and add 3mL sodium chloride-hydroxylamine sulfate solution to reduce excess $KMnO_4$. Add 27.5mL DI water and shake well. Follow the analysis procedure below (Section 10). If additional volume(s) of $KMNO_4$ were added, compensate for the addition(s) by adding less DI water so that the final volume will remain constant.

9.3 Fish and Crustaceans
Blanks and laboratory control standards should be treated identically. All reagents that are added to the samples should be added in the same ratios to the blanks and lab control standards. Fish are calculated on an "as is" basis.

9.3.1 Weigh between 1.0g and 1.2g of the sample and place into a 125-mL glass bottle.

9.3.2 Add 2mL H_2SO_4 and 0.5mL HNO_3 to each sample and digest in the waterbath or heating block for 30min at 80°C or until the tissue is completely dissolved.

9.3.3 After samples are cooled, add 7.5mL of $KMnO_4$ (more $KMnO_4$ may be added if required), 4mL potassium persulfate solution, 25mL DI water and put samples back into water bath for an additional 90min at 30°C.

9.3.4 Remove and cool. Add 3mL hydroxylamine sulfate solution to neutralize excess $KMnO_4$. Add 26mL DI water and shake well. If additional volume(s) of $KMNO_4$ were added, compensate for the addition(s) by added less DI water so that the final volume will remain constant.

10.0 ANALYTICAL PROCEDURE

10.1 Initial startup of the instrument

10.1.1 Before analysis begins inspect the system (pump tubes, mixing coil, gas/liquid separator) to see if any parts need to be cleaned or replaced.

10.1.2 Replace the drying tube with a freshly packed drying tube, making sure that the magnesium perchlorate is not packed too tightly. The vapors must be able to pass freely through the drying tube. Alternatively inspect the pre-made drying tube from Leeman Labs (120-00281-1) for discoloration and clean or replace as needed. Caution should be used if moisture is visible in the tubing that follows the drying tube

10.1.3 Fill the rinse tank with rinse water.

10.1.4 If the lamp is not already on and warmed up, turn on the lamp. The lamp must warm up for a minimum of 2 hours.

10.1.5 If the lamp is already on and warmed up, make sure the platens are tight and turn on the pump Allow a minimum of 20 minutes of pump time for the pump tubes to break in each day.

10.1.6 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle. Allow the reagent to reach the sample stream before starting an autosampler run

10.1.7 Fill the stannous chloride reagent bottle with stannous chloride solution. Switch the reagent line from rinse to the stannous chloride reagent bottle.

10.2 Autosampler setup

10 2 1 Fill the standard tubes with the appropriate standards for the protocol being followed.

10.2.2 Fill the labeled sample test tubes with the samples and calibration verification standards in the applicable order. An example order is as follows:

ICV - Initial Calibration Verification Standard
ICB - Initial Calibration Blank
Detection limit standard
9 SAMPLES
CCV - Continuing Calibration Verification Standard
CCB - Continuing Calibration Blank
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

The preparation blank will be analyzed first The LCS will follow immediately after the preparation blank. The samples, matrix spikes, and duplicates will then follow with a maximum of 10 analyses between CCVs/CCBs. **All samples and control samples must be labeled with the corresponding batch ID.**

10.2.3 Enter the sample/QC IDs into the autosampler table giving each rack a unique name.

10.2.4 Load the rack(s) onto the autosampler.

10.3 Calibration of the mercury analyzer.

10.3.1 Call up the required protocol Open a new data folder.

10 3.2 Go to CALIBRATION, RESET, and reset the calibration for a new calibration.

10.3.3 Go to CALIBRATION, STANDARDS, and insure that calibration standards are entered at the proper concentrations.

10.3.4 Analyze the standards, beginning with standard 1 (Blank), proceeding from lowest to highest concentration.

10.3.5 When all calibration standards have been analyzed, go to CALIBRATION, LINE CALIBRATION. If calibration is within acceptable limits (correlation > 0.995) accept the linear calibration and print the calibration curve.

10.4 Sample analysis

10.4.1 Go to AUTOSAMPLER, SETUP Enter the Rack ID (s) and the cup numbers to be analyzed.

10.5 If the concentration of a sample is above the calibration range of the Hg analyzer, the sample digestate must be diluted and reanalyzed. The amount of digestate needed to prepare the desired dilution is determined from the following equation.

$$V(digest) = \frac{V(fv)}{DF}$$

where

V(digest) = volume of sample digestate used to make the dilution (mL)
V(fv) = final volume of diluted sample (mL)
DF = dilution factor

Samples should be diluted with digested blank solution

10.5.1 The dilution factor is calculated as follows:

$$DF = \frac{V(fv)}{V(digest)}$$

where

V(digest) = volume of sample digestate used to make the dilution (mL)
V(fv) = final volume of diluted sample (mL)
DF = dilution factor

10.5.2 If a sample exceeds the calibration range of the instrument by a factor of 5 or more the samples should be re-digested and reanalyzed with a smaller amount. This would be a good check for possible positive bias of the sample by incomplete digestion of organic compounds. Initial weights or volumes of <0.2g or <1.0mL should be avoided if possible so that a representative sample can be achieved If, due to the level of mercury in the samples, greater dilutions are required, consult with your supervisor for further instructions.

Carryover from high concentration samples usually affects only the next one to two samples in the sequence. The two samples following an off-scale sample that is greater than 10µg/L must be reanalyzed to verify the presence or absence of mercury and the quantitation of mercury It is the responsibility of the analyst to clearly demonstrate that all mercury results are accurate and free from carry-over contamination.

10.6 Post Digestion Spike (Analytical Spikes)
The post digestion spike is performed to verify that samples of similar matrix types are free from interferences from each batch spiked after the preparation/digestion.

10.6.1 Select at least one sample from within a batch for the post digestion spike

10.6.2 Add a known volume of a spiking solution to a known volume of sample digestate. It is suggested that the volume of spiking solution be 1% of the volume or less of the digestate to minimize the effects of volume on the post-digestion spike. The post spiking solution is prepared at a concentration that will yield a spike concentration at or near 2 times the RL when the sample digestate is spiked.

The following equation can be used to determine the volume of spiking solution required: where

$$V1 = \frac{C2 \otimes V2}{C1}$$

C_1 = concentration of spiking solution (mg/L)
V_1 = volume of spiking solution (mL)
C_2 = desired concentration of post digestion spike (mg/L)
V_2 = volume of sample used for post-digestion spike (mL)

10.6.3 Analyze the spiked aliquot and an un-spiked aliquot.

10.6.4 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \otimes 100$$

where

Cps = concentration of post digestion spike (µg/L)
Cs = concentration of un-spiked sample (µg/L)
C2 = theoretical concentration of spike (µg/L) (See 10.2.5.2)

10.6.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 85-115% recovery.

Result of Post Digestion Spikes	Action
Within 85-115% limits	None
>115% recovery	Repeat analysis Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample.
<85% recovery but >50% recovery	Analyze all associated samples by single point method of standard addition and quantify by using MSA. Or qualify all associated samples on report. If sample concentration is less than the IDL, respike (to check for a spiking error), reanalyze, and re-evaluate.
<50% recovery	Dilute digestate and repeat spike. Analyze all associated samples by single point MSA.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high.

10.6.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative absorbance. The first point in the MSA (un-spiked sample) should be greater than or equal to zero absorbance or the magnitude of the negative response should not exceed the reporting limit. Use good judgement when evaluating data where the absorbances are negative. The digestate should be diluted and reanalyzed to determine the extract of the matrix interferences.

10.7 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, Vx, are taken. One aliquot is spiked with a known concentration, Cs. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The absorbance of both aliquots are measured and the sample concentration, Cx, is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S1 = absorbance of the spiked aliquot
S2 = absorbance of the un-spiked aliquot

10.8 Serial Dilution Check
A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

10.8.1 Select one sample per batch for a serial dilution analysis. Analyte concentration should be ≥25 times the instrument detection limit.

10.8.2 Dilute the digestate by a factor of 5 and analyze the dilution using the same procedure used for the unspiked aliquot.

10.8.3 Compare the results of the diluted and undiluted aliquots of sample digestate.

10.8.4 If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than ±10% matrix interference should be suspected and the batch post-digestion spikes should be evaluated.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Aqueous and Leachate Samples

The concentration of mercury in liquid samples is routinely reported as µg/L and is calculated as follows:

$$C(sample) = C(curve) \otimes DF$$

where

C(sample) = concentration of sample (µg/L)
C(curve) = concentration from curve (µg/L)
DF = dilution factor

The RL(in µg/L) is calculated as follows:

$$RL(sample) = RL(lqm) \otimes DF$$

where
RL(sample) = reporting limit of sample (µg/L)
RL(lqm) = reporting limit from LQM (µg/L)
DF = dilution factor (The RL in the LQM assumes that DF=1)

The reporting limit (RL) may also be reported in mg/L. Results in mg/L are reported by dividing the result in µg/L by 1000.

11.2 Soil/Solid Samples

The concentration of mercury in soil and solid samples is routinely reported as mg/kg on a dry weight basis and is calculated as follows:

$$C(sample) = C(curve) \otimes \frac{F \otimes DF}{W \otimes solids} \otimes \frac{1mg}{1000ug}$$

where
C(sample) = concentration of sample (mg/kg dw)
C(curve) = concentration of digest from curve (µg/L)
F = final volume of digest (L)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

The reporting limit (RL) for soil/solid samples is calculated as follows:

$$RL(sample) = RL(lqm) \otimes DF \otimes \frac{1.0g}{W \otimes solids}$$

where
RL(sample) = reporting limit of sample (mg/kg dw)
RL(lqm) = reporting limit from LQM (mg/kg)
W = weight of sample digested (kg)
solids = (percent solids)/100
DF = dilution factor

RL (LQM) is based on a 1-gram sample with a percent solids of 100 (solids =1).
This equation assumes that all digests are taken to the same final volume as the standards

12.0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 SOP AN02: *Analytical Batching and Evaluation of QC Data* and the SOP Summary provide guidance on evaluating QC and sample data. This guidance, including corrective actions, is summarized in Appendix A.

SOP AN02 contains the equations for the evaluation of the QC samples for accuracy and precision as well as corrective actions.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP CA85: *Nonconformance and Corrective Action Procedures*. CA85 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP CA92: *Procedure for Initial and Continuing Analyst Demonstration of Capability*.

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL)*.

14.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

14.1 Pump tubing: Inspect daily and replace as needed.

14.2 Standard Autosampler Cups: Clean daily and replace as needed.

14.3 Drying Tube: Repack daily, or more often if needed. Alternatively use the drying tube from Leeman Labs (120-00281-1) and clean and replace as needed.

14.4 Mixing Coil/Gas-Liquid Separator: Inspect weekly, clean and replace as needed.

14.5 Sample Probe: Inspect monthly, clean and replace as needed.

14.6 Mercury Lamp: Clean or replace as needed.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal*. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

Excess samples, reagents, and standards must be disposed in accordance with SOP CA70: *Waste Management*.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system

- Excess oil samples – Transfer to waste department for storage/disposal

16.0 REFERENCES

STL Savannah's *Laboratory Quality Manual (LQM),* current revision

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (SW-846 Update III).

Methods for Analysis of Water and Waste; U S. EPA Office of Research and Development: Cincinnati, OH, March 1983

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

Appendix A contains an SOP Summary which includes:

- Collection, preservation, and HT summary
- Analytical Sequence
- QC Criteria Summary

Appendix A - SOP SUMMARY

Collection, Preservation, and Hold Times

Container	**Aqueous:** Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid. **Soils:** Minimum 250mL plastic or glass bottle with a plastic or Teflon-lined lid. If other metals are being tested, the aliquot form mercury may be taken from the same container
Preservation	**Aqueous:** HNO3 to pH <2 in the field If dissolved mercury is required, filter the samples before preservation. **Soils:** No chemical preservation required
Storage	**Aqueous:** Room temperature if properly preserved **Solids** should be stored at 4°C (<6°C , but not frozen) from collection until preparation.
Hold Time	**Aqueous and Soils:** Aqueous and Soils: Samples must be analyzed within 28 days of collection.

Wastes are treated in the same manner as soils.

ANALYTICAL SEQUENCE	
Instrument Startup	Turn on the mercury analyzer according to the instrument manufacturer's recommendations. Allow the mercury lamp Proper warm-up time. Inspect and change pump tubes and drying tubes as needed Check and align lamp and cell According to the instrument manufacturers recommendations.
Initial Calibration	Beginning with the blank, calibrate with the blank and 5 standards. One standard must be at or below the RL.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the analysis run. The ICV Solution must come from a source other than the calibration source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with a concentration at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run. The CCV and ICV may be the same solution. Analyze a calibration blank after every CCV.
Detection Limit Check Solution	At the beginning of the analysis run, verify the accuracy at the RL by analyzing a standard with a concentration at or below the required RL.
Post Digestion Spikes/Serial dilution	At a minimum of once per analytical batch, verify the absence of matrix interference by analyzing a post digestion spike and a serial dilution.

Appendix A - SOP SUMMARY

QC CRITERIA

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 blank and 5 standards Correlation > 0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	SW846 = within ± 10% 245.1 = within ± 5%	Recalibrate
Continuing Calibration Verification Standards (CCV)	At the beginning and end of the analysis and every 10 samples.	SW846 = within ± 20% 245.1 = within ±10%	Terminate the analysis, correct the problem and reanalyze all samples since the last compliant CCV.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the required RL.	SW846 = terminate the analysis, Correct the problem and reanalyze all samples since the last compliant CCB.
RL standard (detection limit standard CRA)	After every calibration but not before the ICV.	50-150% of true value	Recalibrate.
Laboratory control sample (LCS)	One per batch of twenty or fewer samples	LQM Limits	Redigest and reanalyze batch
Preparation Blank - SW846	One per batch of twenty or fewer samples	Result < required RL.	Re-digest and reanalyze batch (if sample result >20X the blank, the sample does not have to be re-digested/reanalyzed)
MS/MSD - SW846	One set per batch of twenty or fewer samples	%Rec = 80 – 120% %RPD = < 20%	Flag and report data
MS – 245.1	MS added to a minimum of 10% of samples	%Rec = 70 - 130%	Flag and report data
Serial Dilution Analysis (1+4 dilution)	One per batch of twenty or fewer samples	If sample is at least 25 times the instrument detection limit the serial dilution, corrected for the dilution factor, should agree within +/- 10% of the undiluted sample. (Section 10.8)	Evaluate the post-digestion spike.
Post Digestion Spikes	One per batch of twenty or fewer samples	%Rec = 85 - 115% (Section 10.6)	Check for interference source and reanalyze samples, dilute all samples, or analyze all samples by MSA.

CHLORINATED HERBICIDES

(Methods: EPA 515.1, 615, and SW-846 8151A)

Approval and Signatures

Quality Assurance Manager:	Andrea Teal	Date: March 21, 2005
	Andrea Teal	
Laboratory Director:		Date: March 21, 2005
	Benjamin Gulizia	
EH&S Officer:	Ernest B. Walton	Date: March 21, 2005
	Ernest Walton	

SOP REVISION SUMMARY

Revision Type:

___Minor _X_ Significant ___Complete Re-write ___New SOP

Summary of Changes Since Previous Revision:

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information
- Updated the type of columns used in this procedure
- Added information on herbicide standards and correction factors to Section 8
- Revised the GC parameters in Section 10
- Removed the option to use internal standard calibration for initial calibration of the GC; not performed
- Removed information about the RL standard, no longer used
- Added quality control, method performance, preventative maintenance, and waste management information
- Revised Retention Times for target compounds listed in Appendix A
- Revised GC conditions in Appendix A
- Replaced calibration standard tables in Appendix B
- Updated the Laboratory Performance Solution Criteria in Appendix C, also included in Method Modification in Section 2

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedures used to determine the concentration of chlorinated herbicides in various matrices. Appendix A contains an example of the retention time order for the herbicides, Appendix B provides examples of the calibration standards routinely analyzed, and Appendix C contains a summary of the method QC requirements for Methods 515.1, 615, and 8151A.

Method	Routine Matrices
515.1	Drinking water
615	Water and wastewater
8151	Water, groundwater, soils, solids, wastes, leachates

1.2 The reporting limit (RL), the method detection limit (MDL), and the accuracy and precision limits are listed in the Laboratory Quality Manual (LQM) prepared by and for STL Savannah.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Environmental samples are prepared using the procedures outlined in SOP EX45*: Extraction of Chlorinated Herbicides.* The extracted methyl derivatives are analyzed by a GC equipped with dual capillary columns (different phases) connected to dual electron capture (EC) detectors, allowing simultaneous detection and confirmation of the target compounds. Quantitation may be performed using the external or internal standard calibration technique.

2.2 GC/MS confirmation can also be employed if analyte concentration is sufficiently high or if the sample extract is concentrated to an appropriate final volume. The esterified extract must be used for the GC/MS confirmation – do not use the 8270 extract.

2.3 Method Clarifications/Default Procedures

Elimination of Calibration Points: When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

Bracketing Sample Extracts: The default procedure for continuing calibration verification for the 8000-series methods is to bracket samples by CCV standards (before and after) if external standard calibration is used and ***not*** to cap the sequence (run CCV after the samples) if internal standard calibration is used ***unless*** noted in the client QAPP or in an STL pre-project plan. The internal standard provides verification information on the sensitivity and retention time stability of the instrument and verification of acceptable injections of the sample extracts. See Appendix C for summaries of the analytical sequences.

Grand Mean: The "grand mean" is used to evaluate calibration data according to the provisions of SW-846 Method 8000B and Sections 10.3 and 10.4 of this SOP.

Dilutions: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

2.4 This method is based on the guidance in SW-846 Methods 8000B, 8151A, 40 CFR 136 Method 615, and EPA method 515.1.

2.4 Method Modification:

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). The laboratory does not report 4-Nitrophenol or 3,5-Dichlorobenzoic acid as these analytes are not regulated drinking water analytes; therefore, only the dinoseb sensitivity check is required by this SOP.

2.5 Definitions – Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Glassware should be scrupulously cleaned and solvent-rinsed in accordance with SOP AN60: *Glassware Cleaning Procedures* to minimize artifacts and/or elevated baselines in gas chromatograms. Any vessel that comes in contact with the extract is a potential source for contamination. Method blanks that are extracted and analyzed with each batch of samples will provide clues to the source of contamination from the glassware and reagents.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The sample may require dilution prior to analysis to reduce or eliminate interferences. The extraction procedure SOP EX45 has several steps that are designed to eliminate or minimize interferences due to matrix. The extract is diluted as needed for data analysis. If a cleanup is used, the method blank and lab control standard must also be subjected to the cleanup.

4.3 Injection port maintenance is very important for the consistent detection of the reactive herbicides such as dinoseb.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to Appendix C for a summary of the sample collection, storage, and preservation requirements.

6.0 APPARATUS AND MATERIALS

6.1 Gas chromatograph (GC), temperature programmable, equipped with dual electron capture (EC) detectors and a compatible autosampler

6.2 Data system compatible with the GC, with appropriate software or integration capabilities

6.3 The following column pairs are recommended. Other columns/phases may be used if the calibration and QC criteria are met and adequate separation of the target compounds is achieved.

J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film

6.4 Microsyringes: appropriate volumes

6.5 Volumetric flasks: Class A, appropriate volumes

6.6 Autosampler vials, septa, and caps: compatible with the autosampler

7.0 REAGENTS

The preparation of reagents must be performed in accordance with SOP AN44: *Reagent Traceability.*

Hexane - pesticide grade or equivalent, for preparation of standards

8.0 STANDARDS

8.1 The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Standard Material Traceability.* General guidance on the preparation of standards is given in SOP AN43: *Standard Preparation.*

8.2 The lab should purchase certified solutions from STL approved vendors, if available. The lab should prepare standards from neat materials only if a certified solution is not available. See SOP AN43 for guidance for standard preparation.

8.3 Herbicide standards are purchased as methyl esters; therefore, the concentration of the standard must be corrected to the free acid concentration. This will eliminate the need to correct the final concentration of the sample. The correction factors are given in Appendix D.

8.4 Calibration Standard Recipes

The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. The lowest level calibration standard should be at or below the equivalent of the reporting limit as defined in the LQM or client QAPP. The remaining standards will define the working range of the analytical system. Appendix B contains example recipes of the calibration levels for the routinely determined herbicides.

9.0 SAMPLE PREPARATION

The sample preparation and cleanup procedures are described in SOP EX45

10.0 ANALYTICAL PROCEDURE

10.1 Gas Chromatograph Operating Conditions

The instrument conditions listed in this section are for guidance. The actual conditions used by the lab must be documented in the instrument maintenance log, data system, or run log. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

10.1.1 Two configurations are routinely used for the analysis of herbicides. A single column may be connected to the injection port or two columns may be connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide

simultaneous detection and confirmation of the target analytes.

10.1.2 Example GC Parameters

Columns:
J&W DB-XLB 30 M x 0.32 mm ID x 0.5 um film
J&W DB-35MS 30 M x 0.32 mm ID x 0.5 um film
Injector:240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~2mL/min (per column) (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
HoldTemp 1:	15°C/min to 200°C
Program Rate 2	80°C/min
FinalTemp :	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

NOTE: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize the analytical system.

10.2 Initial Calibration

Internal or external standard calibration techniques may be employed for the determination of the concentration of herbicides. Pentachloronitrobenzene (PCNB) may be a suitable compound to use as an internal standard

10.2.1 Prepare and analyze the calibration standards. Injector port and column maintenance should be performed on the instrument prior to the analysis of the initial calibration standards. Guidance for establishing the analytical sequence is given in the SOP Summary.

Note that the following offers two options for calibration and quantitation – average CF or regression curve. *Only one needs be chosen per analyte.*

10.2.2 Evaluate the standard chromatograms. Some questions to ask at this point are:

>Is there contamination in the hexane blank? If so, has maintenance been performed on the instrument lately? Has the septum been changed? Is the column properly seated in the injector and detector ports?
>Did all of the standards inject properly? Are there peaks for each of the standards analyzed? Do the patterns look normal?
>Are the peaks symmetrical? Is there tailing or fronting?
>Are the areas of the peaks normal for the sensitivity setting being used?

Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

10.2.3 Evaluate the calibration curve in accordance with SOP AN67: *Evaluation of Calibration Curves.*

10.3 Initial Calibration Criteria:

Method 515.1: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 615: If the relative standard deviation is less than 10% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

Method 8151: If the relative standard deviation is less than 20% for the target compounds in the initial calibration, the calibration is considered linear through the origin and the average calibration factor may be used for quantitation.

The preferred method of quantitation is the average response or calibration factor. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve (Section 10.2.5). The "grand mean exception" described below should be applied to 8151A initial calibrations only in extraordinary circumstances because of the difficulty of maintaining and providing documentation on an on-going basis.

8000-series ICAL grand mean exception:
If one or more compounds exceed the %RSD criteria, the average calibration factors can be used for quantitation if the average %RSD of ALL of the compounds (the grand mean) in the ICAL is less than or equal to 20% and no single compound has a %RSD greater than 60%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

Regression Curve Option: A calibration curve is established for each analyte by plotting the concentration along the x-axis and the corresponding response along the y-axis. If r^2 is greater than 0.99, the curve can be used to quantify samples. For 8000-series methods, a minimum of five points is required for a linear regression, six points for a second order curve, and seven or more for higher order fits. It is recommended to use only linear and quadratic (second order) curves for quantitation. See SOP AN67 for guidance on evaluation of calibration curves.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

10.4 Calibration Verification

Calibration is verified at the frequency given in the SOP Summary. If external standard calibration is used, the following criteria apply to calibration standards analyzed before and after samples. In situations where compounds fail criteria high and no positive hits for the compound(s) failing high are detected, these samples may be reported.

If internal standard calibration is used, the samples do not have to be bracketed (capped) by the analysis of a CCV standard unless specified by a regulatory agency or client QAPP.

10.4.1 Analyze a mid-level standard. The concentration of the verification standard should be varied periodically to evaluate the calibration curve in the lower and upper halves. Tabulate the area of the target analytes and calculate the response factors if using the average RF/CF option. If using the calibration curve option, calculation of the RF is unnecessary.

Calculate the percent drift or percent difference between the initial and continuing calibration in accordance with SOP AN67.

10.4.2 Continuing Calibration Criteria

Response Criteria

If the CCV criterion is not met, another CCV should be analyzed. Repeated failure may be a sign of instrument or standard degradation. If the calibration verification criteria cannot be met, a new initial calibration must be prepared, analyzed, and evaluated.

Method 515.1: If the percent drift or percent difference is less than or equal to 20%, the initial calibration is verified and the average response factor or regression curve can be used for quantitation.

Method 615: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

Method 8151: If the percent drift or percent difference is less than or equal to 15%, the calibration curve is verified and the average response factor or regression curve can be used for quantitation.

8000-series CCAL grand mean exception:
If one or more compounds exceed the %drift or %difference criteria, the average calibration factor or regression curve from the initial calibration can be used for quantitation if the average %drift or average % difference of ALL of the compounds (the grand mean) in the CCV is less than or equal to 15% and no single compound is greater than 45%D.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

All samples analyzed using external standard calibration must be bracketed by acceptable CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is *above* the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Retention Time Criteria
The retention time for the CCV must fall within the daily retention time window as defined in SOP AN66: *Determination of Retention Time Windows for Gas Chromatographic Analyses.*

Internal Standard Response Criteria
If internal standard calibration is used, the response of the internal standard(s) must be within -50% to +150% of the response in the CCV-level standard in the initial calibration sequence. If

the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

10.5 Sample Analysis Sequence

The analytical sequences for the methods are given in the SOP Summary in Appendix C. The default is to exclude QC items (method blanks, LCS, and MS/MSD) in determining the maximum number of extracts in the clock. For 8151A, more than 20 extracts (samples and QC) may be analyzed in a sequence, as long as the 12 hour time frame has not elapsed, but the number of samples (non-QC extracts) may not exceed 20. Note that some client and agency QAPPs may require that the QC items be counted as part of the twenty samples.

10.5.1 The sample extract is injected using the same injection volume used for the calibration standards. Extracts that are known to be relatively clean should be analyzed first. Extracts suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.5.2 If the concentration of target compounds exceeds the working range (defined by the highest standard in the initial calibration), the extract must be diluted in hexane and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve. If the internal standard calibration is used, the concentration of the internal standard in the diluted extract must be the same as in the calibration standards.

NOTE: Unless otherwise specified by a client or QA plan, results from a single dilution are reportable as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client.

For clients who demand lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 the dilution factor with the highest target in the upper half of the calibration curve. For example, a sample analyzed at a DF of 50 resulting in a hit in the upper half of the calibration curve would be reanalyzed at a DF of 5 to provide lower detection limits to the client. Project managers and lab staff must work together to balance client satisfaction with productivity.

10.5.3 Occasionally, situations may arise where part of the chromatogram is obscured by large non-target peaks or matrix interferences (short, wide, peaks that are not well resolved). In these situations, it is permitted to report a lower RL for the target compounds that are not affected by the non-target or matrix interference and perform a dilution only for the target compounds that are affected. This anomalous situation must be discussed with the project manager and section supervisor prior to reporting the results and noted in the case narrative or anomaly report. Again, project managers and lab staff must work together to balance client satisfaction with productivity.

10.6 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data Chromatographic Analyses.*

11.0 DATA ANALYSIS AND CALCULATIONS

Methyl ester herbicide standards must be corrected to the free acid concentration. This is performed by comparing the molecular weight of the methyl ester that of the acid ton determine a correction factor. Appendix D gives the molecular weights of the acids and esters. It also lists the correction factors and illustrates how to perform the acid-ester correction.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP AN66: *Determination of Retention Time Windows and Evaluation of Retention Time Data for Chromatographic Analyses* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

11.1 Compounds of Concern

Dalapon - this compound elutes very early in the run and may be subject to interference from co-eluting compounds and from artifacts from the extraction process.

MCPA and MCPP - these compounds have very low response in comparison to the other herbicides.

Dinoseb - this compound can be lost in the extraction process (hydrolysis step) but also may be lost if the injection port is not frequently and properly maintained.

11.2 Manual integrations must be documented in accordance with SOP AN65: *Manual Integrations.* Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.3 The judgement and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?
- Is the internal standard present at the correct retention time and response (-50% to150% of the response in the associated CCV)? Are the surrogates present at the expected RT or have they shifted?

Qualitative analysis

Identification of the surrogates and target compounds is based on retention time. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. The analyst should also note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target

compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect". This may only be performed when the RT of the internal standard and surrogates are at their respective retention times and there is little or no evidence of matrix interferences. If there is doubt as to whether the peak can be excluded or not, the default procedure will be to report the peak as the target compound unless another technique (for example, GC/MS) is used to determine that the target compound is not present.

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks, which have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are handled using dilutions as detailed above (Section 10.5.2).

Evaluate the internal standard (if used) and the surrogates to check for shifts in retention times and to evaluate the surrogate recovery. The recovery criteria for surrogates are given in the LQM.

Internal Standard Criteria

The internal standard must be within the retention time window defined by the associated CCV. The response of the internal standard(s) must be within a range +/–50% of the response of the internal standard in the associated CCV.

If sample matrix interferences preclude the use of internal calibration for a sample extract, two options should be considered:

1) dilute the extract to minimize or eliminate the interference
2) use external standard calibration to quantify the target and surrogate compounds (if external standard calibration is used, all calibration requirements, including a capping standard, must be met - see Appendix C for the external standard sequence).

Surrogate Criteria

DCAA is used as the surrogate for herbicide analysis. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance in the table in Section 11.1.3.

NOTE: If the recovery of the surrogate(s) is above the upper control limit and no target compounds are detected in the sample, results may be reported. Refer to SOP AN02 regarding this issue.

Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

If the concentration is below the lowest calibration standard or MDL (if the sample is being evaluated for "J" results), the reporting limit (RL) for that compound is calculated (Section 11.2). The RL is calculated for all target compounds that are not detected on the primary analytical column. Peaks over-range are handled using dilutions as detailed above (10.5.2).

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range with the %RPD >40, the analysis at a dilution is not necessary.

If the result for a target is above the reporting limit (RL) on the primary column, evaluate the confirmation column. Use the retention time window calculated using the CCV as guidance for the identification of the target compounds. Note shifts in the retention times of the surrogate compounds or internal standard(s) to help gauge possible shifts in the RT of the target compounds. If, in the professional judgement of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

If the target compound is detected on the confirmation column, the concentration of the target compound is calculated and compared to the result from the primary column. The relative percent difference is calculated:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

If the relative percent difference is less than or equal to 40%, the presence of the target compound is confirmed and the higher concentration is reported.

NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A larger relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. Flag the result to note the disparity (P flag) between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

The default guidance in this table assumes the following:
1) the retention time and response of the internal standard(s) are within acceptance criteria with little or no shift in RT
2) surrogate recovery meets the acceptance criteria and peaks fall within the middle of it's retention time window with little or no shift in RT
3) the peak identified as the target falls in the middle of the retention time window for that compound

Default Guidance for Evaluation of Surrogates and Target Compounds in Samples, LCS, and MS

PEAK INFORMATION	COLUMN 1	COLUMN 2	%RPD	REPORT
No peak present	No peak		NA	<RL
		No peak	NA	If compound is a surrogate, re-extract. If sample is LCS, re-extract.
Peak present at RT	<E	<E	<=40%	Report highest
	<E	<E	>40%	Report result most appropriate for sample matrix. Use lowest result as default. Flag with "P"
Peak present at RT	>E	<E	<=40%	Dilute extract to get both results within the calibration curve.
	<E	>E		
	>E	<E	>40%	Report lowest result and flag with "P"
	<E	>E		No dilution required.

E = highest point in curve above which results are flagged as "E". The concentration range for target compounds is RL or MDL to E. Flag results <RL but >MDL as "J". Report result less than MDL as <RL.

MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data are flagged.

11.4 Identification "Tools"

Analysis by GC/MS (scan or SIM) may be used to confirm the presence of the target compounds (see SOP SM06: *Guidelines for SIM Analysis by GC/MS.)*

11.4.1 Relative Retention Time

The retention time of a surrogate compound or internal standard provides useful information about the stability of the GC system. If the surrogate RT has not changed, it is probable that the target analytes RTs have not changed. The relative retention time can help the analyst to evaluate a peak:

$$RRT = \frac{RT_{target}}{RT_{surrogate}}$$

The relative retention time will remain fairly constant under the same GC conditions. The expected retention time of the target can be estimated from the RRT and the RT of the reference (in this case, the surrogate):

$$RT_{target} = RRT \times RT_{surrogate}$$

The analyst must be alert for the presence of matrix interferences and evaluate the data on both columns before making an identification. Another useful tool that employs a similar idea to the RRT is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.4.2 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and

with consultation with the GC supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a 2-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the GC supervisor or other manager with GC experience.

12. 0 QUALITY CONTROL AND DATA ASSESSMENT

12.1 The analytical batch is discussed in SOP AN02: *Analytical Batching and Evaluation of QC Data*, and these criteria are summarized in the SOP Summary included in Appendix C. Calculation of QC data is also given in SOP AN02.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the current revision of the Laboratory Quality Manual prepared by and for STL Savannah.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP *CA92: Procedure for Initial and Continuing Analyst Demonstration of Capability.*

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP CA90: *Procedures for the Determination of Method Detection Limit (MDL).*

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to SOP AN53: *Maintenance Procedures for Laboratory Instrumentation* for routine preventive maintenance and the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in SOP CA70: *Waste Disposal.* Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Hexane extracts and hexane used to rinse glassware, columns, etc. Transfer to flammable waste containers.

16.0 REFERENCES

STL Savannah's *Laboratory Quality Manual (LQM),* current revision

Severn Trent Laboratories' *Quality Management Plan (QMP), current revision*

Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; including Update III. U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.

Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.

METHOD 515.1: DETERMINATION OF CHLORINATED ACIDS IN WATER BY GAS CHROMATOGRAPHY WITH AN ELECTRON CAPTURE DETECTOR Revision 4.1 Edited by J.W. Munch (1995) R.C. Dressman and J.J. Lichtenberg - EPA 600/4-81-053, Revision 1.0 (1981) J.W. Hodgeson - Method 515, Revision 2.0 (1986) D. J. Munch (USEPA, Office of Water) and T. Engel (Battelle Columbus Laboratories) - National Pesticide Survey Method 3, Revision 3.0 (1987) R.L. Graves - Method 515.1, Revision 4.0 (1989) **NATIONAL EXPOSURE RESEARCH LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OHIO 45268**

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

APPENDIX A – Target Compounds and Retention Times

Compound	RT COL 1	RT COL 2
Dalapon	2.18	2.17
DCAA (surrogate)	10.00	10.12
Dicamba	10.07	10.26
MCPP	10.30	10.31
MCPA	10.45	10.52
Dichlorprop	10.63	10.66
Bentazon	11.42	12.18
2,4-D	10.77	10.89
Pentachlorophenol	11.18	11.15
2,4,5-TP (Silvex)	11.28	11.30
2,4,5-T	11.50	11.62
2,4-DB	11.84	11.94
Dinoseb	11.93	11.87
Picloram	12.38	12.99
DCPA	12.52	12.66

COL1 _ J&W DB-XLB
COL2 _ J&W DB-35MS

Retention Times of Target Compounds with the following conditions:

Injector: 240°C
Detector: 300°C
Carrier Gas Flow: Helium at ~5.3mL/min (pressure at 20psi, constant)
Make-up Gas Flow: Nitrogen at ~60mL/min (per detector)

Temperature program:

Initial Temp:	50°C
Initial Hold:	0.50 min
Program Rate 1	12°C/min to 100°C
Program Rate 2	15°C/min to 200°C
Program Rate 3	80°C/min
Final Temp:	300°C (hold for 1.25 minutes)
TOTAL TIME	15.73 minutes

APPENDIX B - Example Standard Preparation Recipes

Intermediate Calibration Stock Standard

Stock Standard	Concentration (ug/mL)	Volume Added (uL) to final volume of 10mL in hexane	Final Concentration (ug/mL)
Herbicide Methyl Ester Mix	See below *	250	2.5/25/500
Pentachloroanisol	100	250	2.5
DCAA Methyl Ester	100	250	2.5
Picloram Methyl Ester	100	250	2.5
DCPA	1000	50	5.0

* Methyl Esters of MCPA/MCPP at 20000 ug/mL; Dalapon at 1000 ug/mL; 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP, Dicamba, Dichlorprop and Dinoseb at 100 ug/mL.

Calibration Standards

	Cal. Level 1*	Cal. Level 2*	Cal. Level 3*	Cal. Level 4**	Cal. Level 5*	Cal. Level 6*	Cal. Level 7*
Intermediate Calibration Stock Standard	100 uL	200 uL	400 uL	3000 uL	800 uL	1000 uL	2000 uL
Final Concentration	0.025/0.25/5.0 ug/mL	0.050/0.50/10 ug/mL	0.10/1.0/20 ug/mL	0.15/1.5/30 ug/mL	0.20/2.0/45 ug/mL	0.25/2.5/50 ug/mL	0.50/5.0/100 ug/mL

* 10 ml final volume in Hexane
** 50 mL final volume in Hexane

APPENDIX B - Example Standard Preparation Recipes

HERBICIDE MOLECULAR WEIGHTS AND CORRECTION FACTORS

Herbicide acid	MWacid	MWester/ether	Correction factor
2,4-D	221.04	235.07	0.940
Dalapon	142.97	157.00	0.911
2,4-DB	249.09	263.12	0.947
Dicamba	221.04	235.07	0.940
Dichloroprop	235.07	249.09	0.944
Dinsoeb	240.22	254.24	0.945
MCPA	200.62	214.65	0.935
MCPP	214.65	228.67	0.939
2,4,5-TP(Silvex)	269.51	283.54	0.951
2,4,5-T	255.48	269.51	0.948
DCAA	205.04	219.07	0.936
Picloram	241.48	255.51	0.945
Pentachlorophenol	266.35	280.37	0.950

Example Calculation

$$CF(2,4-D) = \frac{Wacid}{Wester} = \frac{221.04}{235.07} = 0.94$$

If the standard is expressed as mass of ester per volume, convert the concentration to he acid form by multiplying by the correction factor (CF).

APPENDIX C – Method Summary

HOLD TIMES

MATRIX (method)	Chemical Preservative/ Storage*	Routine Container	Sample Hold Time	Extract Hold Time
Drinking water (515.1)	80mg sodium thiosulfate per liter; 4°C	1-L amber x 2	14 days	28 days
Groundwater and Wastewater (615 and 8151)	None; 4°C	1-L amber x 2	7 days	40 days
Soils and wastes (8151)	None; 4°C	500-mL glass	14 days	40 days

*Storage temperature is 4°C with control criteria of less than 6°C with no frozen samples

EXTRACTION SUMMARY – detailed description in SOP EX45

Aqueous - adjust 500mL of sample to pH >12 and hydrolyze for one hour; extract with methylene chloride to remove non-targets and discard solvent; adjust aqueous phase to pH <=2 and extract with diethyl ether; concentrate, esterify and dilute to 10mL final volume with hexane

Soils - acidify 30g of sample, mix with acidified sodium sulfate; and sonicate with diethyl ether; hydrolyze with KOH at pH>12 and discard solvent; adjust aqueous phase pH to <=2 and extract with diethyl ether; concentrate, esterify, and dilute to final volume of 10mL with hexane.

ANALYSIS

Dual capillary columns with dual EC; 2-5uL injection into glass tee or y-splitter; external or internal standard calibration

SURROGATE:

DCAA - 2.0ug/L

BATCH QC

Method blank
LCS/LCSD- full target list of single peak analytes
MS/MSD- full target list of single peak analytes

Analytical Sequence

Method 515.1

Initial Calibration Standards
Initial Calibration Verification (ICV)
Laboratory Performance Solution (Daily)
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration
Client samples analyzed until 12 hour clock expires
Calibration Verification standard – vary concentration

Method 615

Initial Calibration Standards
Initial Calibration Verification (ICV)
Client samples analyzed until 24 hour clock expires
Calibration Verification standard – mid-level concentration
Client samples analyzed until 24 hour clock expires

Method 8151A

Initial Calibration Standards
Initial Calibration Verification (ICV)
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration
20 client samples or 12 hours
Calibration Verification standard – mid-level concentration

The sequence continues until all samples have been analyzed or until the calibration verification fails the acceptance criteria. All sample extract analyses must be bracketed by acceptable verification standards if external standard calibration is used; if internal standard calibration is used, capping of the sequence by a CCV standard is not required unless specified in an agency or client QAPP. The default procedure is not to count the QC items in the 20 sample extracts that may be analyzed in the clock; i.e.; the number of sample and QC extracts may exceed 20 but the total number of sample extracts may not exceed 20 and all extracts (samples and QC) must be analyzed within the 12-hour clock.

Laboratory Performance Solution Criteria (EPA 515.1)

Test	Analyte(s)	Concentration (ug/mL)	Criteria
Sensitivity	Dinoseb	0.004	S/N >3

S/N = a ratio of peak signal to baseline noise.

EPA Method 515.1 requires the analysis of a Laboratory Performance Check (LPC). In addition to the sensitivity check for dinoseb, the EPA 515.1 LPC includes checks for chromatographic performance (using 4-Nitrophenol) and column performance (using 3,5-Dichlorobenzoic acid and 4-Nitrophenol). Since the laboratory does not report these analytes, only the dinoseb sensitivity check is required by this SOP.

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration- 5-point minimum with lowest point at RL	Prior to sample analysis or when CCV fails	1) 515.1 %RSD of each target <=20% or r^2 >0.99 2) 615 %RSD of each target <=10% or r^2 >0.99 3) 8151 %RSD of each target <=20% or r^2 >0.99 (see Section 10.2 for 8000-series "grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and analyze standards
Initial calibration verification (Second Source ICV)	After Initial Calibration	515.1: Percent difference <=20% 615/8151: Percent difference <= 15%	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Laboratory Performance Check Solution	Daily, prior to sample analyses	See criteria in previous section.	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	515.1/8151: After every twenty sample analyses (or 12 hours) and at the end of the sequence 615: After every twenty sample analyses (or 24 hours)	515.1:Percent difference or drift <= 20% 615/8151: Percent difference or drift <= 15% (see Section 10.3 for 8000-series"grand mean" exception)	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze standard(s) -Remake and reanalyze standard(s) -Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in LQM	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Lab control sample (LCS)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. -Check calculations and reanalyze -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze

QC CHECK	FREQUENCY	ACEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix spike (MS) and matrix spike duplicate (MSD)	Per batch	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Surrogate	All samples, method blanks, and QC	Recoveries within LQM limits	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze
Initial Demonstration of Capability (IDOC)	Initially and when new analysts trained	Evaluate in accordance with SOP 92 and method criteria	Repeat test for analytes that fail criteria
Method Detection Limit (MDL)	See SOP CA90	Evaluate in accordance with SOP CA90	Evaluate in accordance with SOP CA90
Retention time window determination	See SOP AN66	See SOP AN66	See SOP AN66